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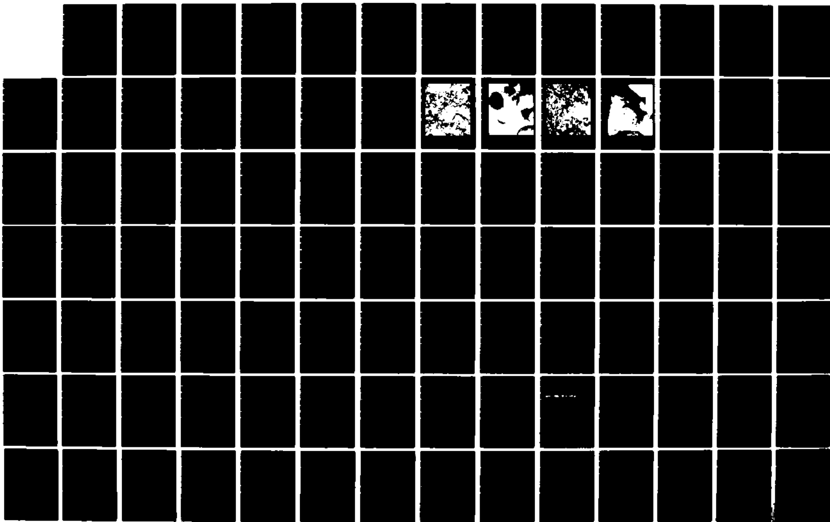

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AD-A170 085

MTL TR 86-18

A PROCESS STUDY FOR MANUFACTURING ULTRA-FINE TITANIUM DIBORIDE POWDER

May 1986

PETER T. B. SHAFFER
Advanced Refractory Technologies, Inc.
699 Hertel Avenue
Buffalo, New York 14207

FINAL REPORT

Contract DAAG46-83-C-0171

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Watertown, Massachusetts 02172-0001

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SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER MTL TR 86-18	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) A PROCESS STUDY FOR MANUFACTURING ULTRA-FINE TITANIUM DIBORIDE POWDER		5. TYPE OF REPORT & PERIOD COVERED Final Report 8/26/83 to 6/25/84
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) Peter T. B. Shaffer		8. CONTRACT OR GRANT NUMBER(s) DAAG46-83-C-0171
9. PERFORMING ORGANIZATION NAME AND ADDRESS Advanced Refractory Technologies, Inc. 699 Hertel Ave Buffalo, NY 14207		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS D/A Project: 1L263102D071
11. CONTROLLING OFFICE NAME AND ADDRESS U.S. Army Materials Technology Laboratory ATTN: SCLMT-ISC Watertown, MA 02172-0001		12. REPORT DATE May 1986
		13. NUMBER OF PAGES 132
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		15. SECURITY CLASS. (of this report) Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Titanium diboride
Synthesis chemistry
High temperature
Boron carbides
Titanium oxides

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

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ABSTRACT

Advanced Refractory Technologies, Invc., of Buffalo, NY has demonstrated the feasibility of utilizing a rotary high temperature carbon tube furnace to form reasonably pure titanium diboride powder. The reaction used was the carbothermic reduction of boron carbide in the presence of highly reactive titanium dioxide. The resultant titanium diboride powder was characterized by X-ray diffraction analysis and microscopic observation. The powder possessed a mean specific surface area of 0.5 square meter per gram. The rate of powder formation through use of the rotary kiln process was approximately five kilograms per hour.

Experimental trials to form fully dense titanium diboride by hot pressing were unsuccessful. Further developmental work utilizing the rotary kiln process to attempt to achieve stoichiometric titanium diboride powder of the required purity for successful hot pressing is indicated.

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I SUMMARY

The objective of this research program was to determine whether or not the new proprietary high temperature furnace technology of Advanced Refractory Technologies could be used for, or adapted to, the production of high quality, fine, titanium diboride powder. Additionally it sought to learn whether or not such fine titanium diboride powder would lend itself to routine hot pressing to dense, monolithic shapes.

The project was successful in both goals. We have produced titanium diboride at a rate of approximately 5 kg per hour on a continuous basis. Powder as produced showed surface areas in excess of 0.5 m^2/g, equivalent to a mean spherical particle equivalent of 2.5μm. The powders so produced were shown to have purities equivalent to commercially available powders.

An as-produced powder, sieved through a 325 mesh screen, was shown to hot-press to over 98% of theoretical density without further treatment.

The effects on powder chemistry and physical properties of variables such as reactant powder composition and size have not yet been evaluated. Neither has the process been fully optimized. Results are, however, such that one can safely state that the Advanced Refractory Technologies furnace technology is well suited to the continuous production of high quality titanium diboride powder.

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II BACKGROUND

Titanium diboride is an interstitial compound of the family of so called "hard metals". It exhibits a unique combination of properties including low bulk density (4520 kg/m^3; 4.52 g/cm^3), extreme hardness (K50g = 3400 kg/mm^2), and extreme refractoriness (mp 2980°C). In spite of these excellent properties, titanium diboride, and to a large extent all of the refractory carbides, borides, nitrides and silicides, has remained an expensive, little used material.

The explanation for this is simple. As presently manufactured, the refractory hard metals are prepared by reacting compacts produced from intimately mixed reactants at extremely high temperatures (in excess of 2000°C). Products from such reactions consist of hard, sintered, brittle and metallic shapes which then must be crushed, ground, and milled to provide powders of sufficient fineness to permit subsequent fabrication into useful artifacts. The batches so produced are small and not infrequently require second, even third, firings to achieve stated chemical specifications.

Each crushing and milling step of these hard materials introduces impurities due to attrition of the grinding media and equipment. Such impurities must be removed prior to use or to refiring.

All of this mechanical comminution, analyses, acid leaching, reblending, and refiring makes the price of the final products prohibitive for all but special applications where either the quantity of material required is small or the application demands certain performance almost without regard to materials costs.

The batch type of production, the highly labor intensive nature of the process, and the comparatively small quantities of material produced seem to rule out the economical mass production of titanium diboride by present technology. What seems to be required is a continuous process in which large quantities of powder could be produced directly in a form suitable for subsequent use (such as hot-pressing) with a minimum of post furnace treatments.

Advanced Refractory Technologies (ART) set out in this program to demonstrate whether the new proprietary high temperature furnace technology was suitable for, or could be modified to, the continuous large scale production of fine titanium diboride powder suitable for hot pressing or other use without extensive post-furnacing treatments.

III INTRODUCTION

Titanium diboride can be formed by any of a number of chemical reactions including:

1) $Ti + 2B \rightarrow TiB2$

2) $TiCl_4 + 2BCl_3 + 5H_2 \rightarrow TiB_2 + 10HCl$

3) $TiO_2 + B_2O_3 + 5C \rightarrow TiB_2 + 5CO$

4) $2TiO_2 + B_4C + 3C \rightarrow 2TiB_2 + 4CO$

Each has advantages as well as limitations. Reaction (1) is the simplest, and is high exothermic but the reactants are not only expensive but potentially pyrophoric. Reaction (2) can lead to products having very high purities since the reactants are distillable, however, not only are the reactants expensive, but theoretical yield is low and the quantity of by-products requiring disposal is large. It is difficult to bring about such gas phase reactions to produce fine powders in production quantities. Reaction (3) is probably the least expensive but losses of volatile boric oxide make control of final stoichiometry difficult.

Reaction (4) represents, we feel, the best choice for large scale commercialization. Reactants are reasonably priced and available in quantities necessary for quantity production. No volatile reactants are present and by-product disposal is simple. Since extra carbon is required in the reaction, low-boron boron carbide may be used in the synthesis. A boron carbide-carbon mixture having only 50% boron can be used. Such low-boron boron carbide powders are easily produced and are comparatively inexpensive.

This is the reaction selected for evaluation.

The program consists of iterative preparations of titanium diboride in which mix compositions were varied, and resulting titanium diboride products were evaluated on the basis of relative degree of conversion, stoichiometry, furnace through-put, crystallinity, X-ray diffraction, and surface area.

The original intent of the program was to include investigations of the effects of time, and temperature of reaction as well as the effects of raw material composition and properties on the reaction and the resulting product. It turned out that this was too optimistic and practically all of the effort was directed toward control of final powder chemistry through mix composition. Such studies will have to wait until further research is initiated, or until it can be made part of a scale-up furnace process optimization.

IV EXPERIMENTAL APPROACH TO THE PROBLEM

The reaction of titanium dioxide with boron carbide and carbon to produce titanium diboride is well understood. (1,2,3) It is the most frequently used reaction in the laboratory to produce experimental quantities of titanium diboride. Although this reaction is somewhat more expensive than the corresponding reaction involving boric acid as a reactant, it is generally selected since the problems associated with volatile boron oxides, and thus the control of stoichiometry are simplified.

Our approach to the problem has involved repetitive reactions in which changes in starting composition were related to final compositions and properties.

These reaction mixes were fired in ART's proprietary high temperature rotary furnace. While time, temperature, atmosphere, etc. were on the original plan as variables to be evaluated in detail, analytical questions forced us to confine our effort largely to attempts to relate initial mix and final product characteristics.

Several experiments were directed toward the refiring of furnace products to carry reactions closer to completion and second firing. This latter is to be differentiated from refiring in that second-firing involves additions to bring the reaction chemistry closer to some prescribed value.

Thirty individual furnace experiments have been completed. Details of each experiment are summarized in Appendix A. Briefly Table I summarizes, on a molar percentage basis the mix composition and as well as those of the products.

Analyses and other characterizations of the various reactants are summarized in Appendix B.

Part of the program involved a comparison of ART produced titanium diboride powder with commercially available materials. These data are summarized in Appendix C. Our analyses are further compared with data quoted in product spec sheets and with laboratory certification where such were available.

V. RESULTS AND DISCUSSIONS

A. PHYSICAL - MORPHOLOGY

Titanium diboride produced by ART's process consists of agglomerates of fine, somewhat rounded, well formed microcrystals (Figures 1 & 2). By heating the reactant mixture to high temperature, maintaining it at temperature only briefly, then quenching the product to below crystallization temperature major crystal growth is prevented from occurring. The brief excursion above crystal growth temperatures is confirmed by the predominance of well formed crystal facets and by the absence of large crystals.

Commercially available TiB_2 powders are produced by crushing and milling larger, sintered bodies resulting from the prolonged high temperature exposure of the initially formed fine powders. It is necessary to expose the larger masses to high temperatures for prolonged periods to insure complete reaction. Similarly the large masses retain the heat and cool slowly further increasing both sintering and crystal growth. The resulting sintered hard, metallic pieces must be crushed and milled. Aside from the introduction of impurities which must be removed prior to subsequent fabrication and use, the resulting powders then consist of irregular angular fragments onto whose surfaces quantities of very fine particles are adhering (Figures 3 and 4).

The directly produced powder clearly has a more uniform particle size distribution, as well as a distincly different morphology.

B. BINDERS

In order to promote reaction, the reactants were intimately mixed by milling them together in a Sweco DM-3 Model vibratory mill using boron carbide cylinders as the grinding media. Due to the nature of fine powders particularly of the materials used in this study (carbon), the milled powders had extremely poor flow characteristics.

To improve the flow characteristics the milled powders were then pelletized using an Eirich high speed pelletizer. The binder system selected initially consisted of 9% corn syrup as a 50% solution in water, with subsequent additions of water to induce pellet formation. The residue from high temperature bake out is a pure, finely dispersed form of carbon which entered the reaction, thus introducing no contamination in the final product.

During this program it was determined that this binder system was not entirely satisfactory. During heat up, if the temperature is not adequately controlled the sugar residue melts, and then begins to froth as water is driven out and the sugar decomposes. Integrity of the pellets can be lost and under the worst case the entire mix coalesces and must be recrushed before use.

In addition there is strong evidence that the amount of carbon residue depends on the conditions under which the sugar is carbonized.

Either of these effects is enough to rule out use of the binder system for long term consideration or for production purposes.

We investigated a number of alternative systems in an attempt to find a binder system which would produce a strong micro pellet and one which would not introduce unwanted residues to the reaction. Ideally the bond phase should not melt below reaction temperatures and be inexpensive.

An inorganic bond has been developed which meets all of these criteria. It was used in later experiments (XXVIII and later). It solved the problem so effectively that the pellets pass through the reaction zone almost intact, so much so that furnace operating conditions have had to be modified to prevent their passage through the furnace without reacting completely.

It is thought that this new binder concept represents a patentable development. Therefore it is not possible to discuss or disclose details about the binder chemistry until patent action is initiated. A follow on report will be issued as soon as this matter has been resolved.

C. ANALYTICAL

Analyses, both titanium and boron, have been subject to question. Taking a total of all of the identified constituents, many of the analyses accounted for less than 95% of the sample. Repeated analyses of the same sample by standard wet chemical techniques showed titanium concentrations varying by nearly 5%. Submission of the same sample to three laboratories gave variations of over 3%. (Table II)

These results, together with unexpectedly high or low values when the experiment was directed in the other extreme, caused concern.

Since boron analyses are a standard certified analysis in ART's nuclear business we approached this internally. We found, simply, that standard fusion conditions are not adequate to bring about complete solution of these perfectly formed microcrystalline powders. Whereas the usual fragmentary titanium diboride powders dissolve readily in the standard fusion, ART produced powders frequently left a residue after fusion.

Later lots of titanium diboride powder (XXVI, 840413 AFR and XXVII, 840416 AFR) were specifically directed toward the formation of a high-boron product. Initial analyses were unexpectedly low, both for titanium and boron. There were reanalyzed using more vigorous fusion conditions. (Table III)

Not only were the results much closer to what we expected, but the mass balance was about an order of magnitude closer to the theoretical unity.

Analyses for titanium were somewhat more consistent, however variation from lab to lab (Table II) were frequently over 3%. Inquiry to the National Bureau of Standards showed that no reference material is available. While it is not strictly a part of this program, we have contacted several laboratories including DOE's Oak Ridge National Labs and have distributed a commercial TiB_2 powder for "round robin" analysis. At the very least we should obtain a concensus and have a reference material for comparison. In as much as the results from these studies are also not yet available they will be covered, and distributed in a future report.

D. REFIRING

It is common practice in the carbide industry to refire and even third-fire powders to produce the desired chemistry. We attempted such an experiment using portions of the TiB_2 produced in this study.

All of the coarse fractions containing unreacted materials and various other products were combined, milled and blended to yield a batch of powder (88kg, 194 lbs.). This was tumbled to homogenize it then analysed (Appendix A). Aside from calcium (0.3%) silicon (0.1%) and iron (0.006%) it contained no non-reacting impurities in excess of 0.006%.

Based on these analyses(C-260,NB IX p.146), it was blended with boron carbide (4.9%), boric acid (3.7%) and sugar binder sufficient to yield a residual carbon of 0.4%. It was then formed into pellets (1-3 mm diameter), dried and refurnaced at 2075°C.

This refired material is summarized as Lot XXVI, Appendix A.

Interestingly the refired material as it came from the furnace was not the usual soft black, lusterless powder, but consisted of hard, sintered nuggets which required significant grinding to convert them into the -325 mesh powder. As normally produced, the first fired powder is so soft that macroscopic agglomerates can easily be crushed between ones fingers.

E. HOT PRESSING

To obtain high density hot-pressed parts from carbides and borides, the powders are routinely milled to particle sizes of the order of 10μm and less. Since the as produced titanium diboride powders showed surface areas corresponding to mean spherical equivalent sizes of 2 - 5μm we submitted a sample of -325 mesh (minus 45μm) powder for hot pressing.

Without further treatment, this powder hot-pressed to a final density of 4.45 g/cm^3 (98.5% of theoretical) using standard conditions. Clearly the small ultimate crystal size has a major effect on the pressibility of the powder. (4)

F. Economics

The continuous nature of the present process for producing titanium diboride powder lends a major economic advantage, compared to present batch processes. In addition producing a fine powder directly largely eliminates all of the crushing and grinding operations as well as most of the final milling. Introduction of impurities is thus greatly reduced. This in turn greatly reduces the need for subsequent acid washing and clean up.

Large quantity continuous production greatly reduces the per pound costs associated with analytical and quality control operations.

While even the relatively modest present production rates of 5 kg/hr. bring with them significant cost savings, the real effect comes when scale up to mass production takes place. We have been asked to prepare a cost estimate for a major on-site production facility, a facility designed to produce in excess of 100,000 kg/yr. The estimate (with deletions of sensitive information) is included as Appendix D. The cost estimate of less than $5 per pound, plus labor charges, is believed to be a realistic estimate.

G. Commercial Materials

Part of this program was to compare the experimental TiB_2 powders with commercially available ones. Characterization of the various commercially available powders showed that some are excellent, while others are next to worthless. (Table IV)

One material, advertised as 99.8% (Atl. Eq. Eng.) contained over 7% impurities (C, O & N) and had at least 3 separate TiX type phases as shown by x-ray diffraction.

A second material (E.S.K.) showed a strong graphite peak by x-ray diffraction, although analysis showed only 1.65% total carbon.

We were discussing TiB_2 with a research group studying TiB_2 for use in cathodes in Hall cells for aluminum production. They commented that seemingly identical lots of TiB_2 gave quite different results. We were supplied with several samples from batches which gave good results as well as bad. These analyses are included for comparison.

It should be noted that two lots of TiB_2 from H. C. Starck showed boron levels far below those reported in the certification analyses. Lot VII-114 showed 23.5%, versus 30.9% reported and lot VII-115 showed 25.5 versus 30.2%. The question of analytical procedure can be ruled out since a sample of TiB_2 from KBI, analyzed in the same lot showed 30.1%.

One should be careful, as shown by all of this, not to accept all product claims at face value. Even coarse materials (Cerac) differ widely from the claimed levels of purity.

VI SUGGESTIONS FOR FUTURE STUDY

The initial goals of this study included evaluation of the effects of variations in starting materials (composition, particle size), in pre furnace treatments (milling, pelletizing), and in time and temperature on the chemistry and properties of the titanium diboride powder produced via the high temperature rotary furnace. Effect of pellet size versus furnace residence time would be among the first for study.

Due to the difficulty in analytical characterization of these powders, and unexplained variations in product chemistry associated with these analytical variations, most of these variations remain unevaluated.

The future study plan remains much the same as that of the present study, with a mjor exception. It has been clearly demonstrated that under proper control, fine high boron titanium diboride powder can be produced directly, and in high volumes by means of the high temperature rotary furnace.

VII CONCLUSIONS

1. The high temperature rotary furnace has been shown to be suitable for the production of titanium diboride powder.

2. As produced, such powders have been shown to have chemistry comparable to that of commercial materials.

3. High surface areas of the as produced powder correspond to mean crystallite sizes less than 5μm.

4. As produced such powders show good hot-pressability.

5. Throughput rates of the order of 5 kg/hr (11 lb/hr) of titanium diboride have been demonstrated together with final boron levels of 30% (Expt XXIV). This is equivalent to a rate of 45,000 kg (100,000 lb) per year per furnace.

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SUMMARY OF FIGURES

1. Titanium diboride powder (ART Lot 840124F) showing loosely bonded agglomerates. Approximate magnification 2000 x.

2. Titanium diboride microcrystallites (ART Lot 840124F). Approximate magnification 25000 x.

3. Commercial titanium diboride powder (H. C. Starck, A Grade) showing predominantly irregular fracture fragments. Approximate magnification 2000 x.

4. Commercial titanium diboride powder (H. C. Starck, A Grade) showing the presence of large fragments onto which fine particles are adhering. Approximate magnification 25000 x.

Figure 1. Titanium Diboride Powder (ART Lot 840124 F) showing loosely bonded agglomerates. Approximate magnification 2000 x.

Figure 2. Titanium Diboride Microcrystallites (ART. Lot 840124 F). Approximate magnification 25000 x.

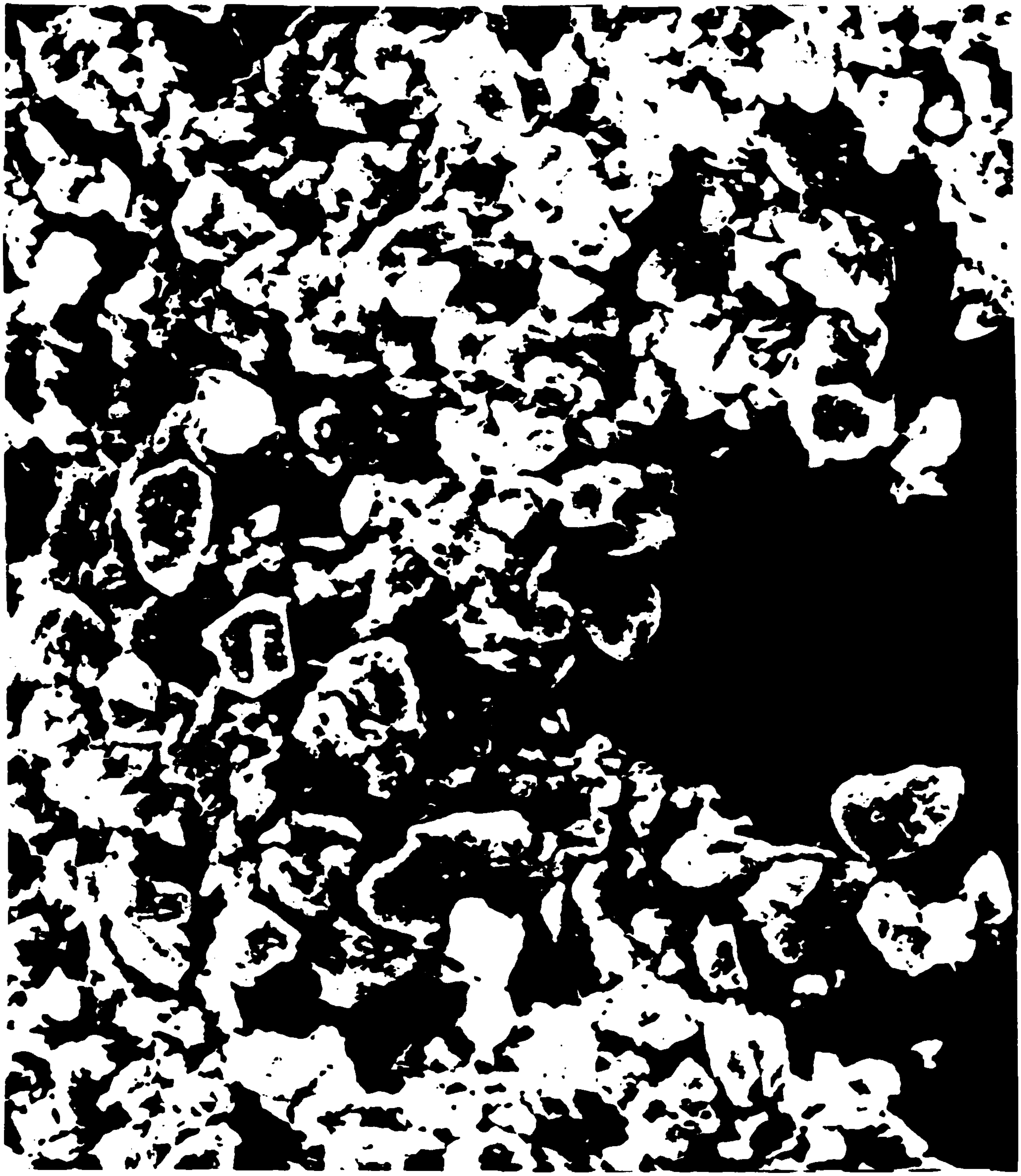


Figure 3. Commercial Titanium Diboride Powder (H. C. Starck, A Grade) showing predominantly irregular fracture fragments. Approximate magnification 2000 x.

Figure 4. Commercial Titanium Diboride Powder (H. C. Starck, A Grade) showing presence of large fragments onto which fine particles are adhering. Approximate magnification 25000 x.

SUMMARY OF TABLES

TABLE I

MOLAR COMPOSITION OF TiB_2 MIXES
AND RESULTING PRODUCTS
(ATOMIC PERCENT)

EXPERIMENT	REACTION MIX					PRODUCT					
NO.	Ti	B	O	C	B/Ti		Ti	B	O	C	B/Ti
I	14.6	31.0	28.4	26.0	2.12		32.3	58.5	0.5	8.6	1.81
II	14.5	28.7	28.8	27.9	1.98	(1)	31.0	54.3	5.6	9.1	1.75
						(2)	32.4	53.2	5.5	8.9	1.64
III	14.5	28.7	28.8	27.9	1.98	(1)	34.0	61.4	1.7	3.0	1.81
						(2)	34.1	61.2	1.7	3.0	1.79
IV	14.5	28.7	28.8	27.9	1.98	(1)	33.1	62.1	2.8	2.1	1.88
						(2)	33.9	61.3	2.7	2.0	1.81
V	14.5	28.7	28.8	27.9	1.98	(1)	34.1	62.3	1.8	1.9	1.83
						(2)	34.2	62.2	1.7	1.9	1.82
						(3)	36.2	60.1	1.8	1.9	1.66
VI	14.6	30.6	29.1	25.7	2.10	(1)	33.5	62.7	2.3	1.5	1.87
						(2)	34.3	61.9	2.3	1.5	1.81
VII	14.6	30.6	29.1	25.7	2.10	(1)	34.1	62.6	0.9	2.4	1.84
						(2)	34.3	62.5	0.9	2.4	1.82
VIII	15.2	31.3	30.3	23.2	2.06	(1)	35.3	61.3	0.6	2.8	1.74
						(2)	35.7	60.9	0.6	2.8	1.71
IX	15.2	31.3	30.3	23.2	2.06	(1)	36.8	60.8	0.4	2.0	1.65
						(2)	36.8	60.8	0.4	2.0	1.65
X	14.8	32.2	29.6	23.3	2.18	(1)	35.7	58.7	0.9	4.8	1.64
						(2)	36.7	57.7	0.9	4.8	1.57
XI	14.8	32.2	29.6	23.3	2.18	(1)	35.4	60.7	0.8	3.1	1.72
						(2)	35.7	60.4	0.8	3.1	1.69

EXPERIMENT NO.	Reaction Mix Ti	Reaction Mix B	Reaction Mix O	Reaction Mix C	Reaction Mix B/Ti	Product Ti	Product B	Product O	Product C	Product B/Ti
XII	14.5	29.0	28.2	28.3	2.00	(1) 34.3	56.7	1.1	7.9	1.66
						(2) 35.7	55.4	1.1	7.8	1.55
XIII	14.5	29.0	28.2	28.3	2.00	(1) 35.5	54.0	2.6	7.9	1.52
						(2) 37.7	52.2	2.5	7.6	1.38
XIV	14.6	29.2	28.4	27.9	2.00	(1) 32.9	62.5	0.6	4.0	1.90
						(2) 35.5	60.0	0.6	3.9	1.69
XV	14.6	29.2	28.4	27.9	2.00	(1) 35.8	59.8	0.6	3.7	1.67
						(2) 37.4	58.3	0.6	3.6	1.56
XVI	14.4	29.5	28.0	28.1	2.05	(1) 32.2	52.2	0.8	14.9	1.62
						(2) 33.4	51.2	0.8	14.6	1.53
XVII	14.4	29.5	28.0	28.1	2.05	(1) 31.1	52.5	0.7	15.7	1.69
						(2) 33.3	50.8	0.7	15.2	1.52
XVIII	15.0	33.3	29.2	22.4	2.22	(1) 38.9	56.2	3.5	1.4	1.45
						(2) 39.6	55.5	3.4	1.4	1.40
XIX	15.0	33.3	29.2	22.4	2.22	(1) 37.0	58.5	2.2	2.3	1.58
						(2) 37.8	57.8	2.2	2.3	1.53
XX	13.7	31.9	26.7	27.7	2.33	(1) 37.3	59.5	2.0	1.2	1.60
						(2) 38.1	58.7	2.0	1.2	1.54
XXI	13.7	31.9	26.7	27.7	2.33	(1) 37.7	59.9	1.8	0.7	1.59
						(2) 38.9	58.7	1.7	0.7	1.51
XXII	14.5	35.6	28.2	21.6	2.46	(1) 36.5	60.0	2.2	1.3	1.64
						(2) 38.8	57.8	2.2	1.2	1.49
XXIII	14.1	31.1	27.4	27.4	2.21	(1) 31.7	61.4	0.5	6.4	1.94
XXIV	13.6	28.9	27.2	30.2	2.13	(1) 34.2	63.2	2.0	0.5	1.85
						(2) 34.1	63.3	2.1	0.5	1.86

EXPERIMENT	REACTION MIX					PRODUCT				
NO.	Ti	B	O	C	B/Ti	Ti	B	O	C	B/Ti
XXV	13.5	31.7	27.0	27.8	2.35	(1) 32.0	62.7	4.9	0.5	1.96
						(2) 32.7	62.0	4.9	0.5	1.89
XXVI	34.1	57.9	4.4	3.6	1.70	(1) 29.9	67.3	0.7	2.1	2.25
XXVII	34.1	57.9	4.4	3.6	1.70	(1) 31.1	66.9	0.6	1.4	2.15
XXVIII	14.3	27.8	28.6	29.3	1.94	(1) 18.2	61.0	2.2	18.6	3.34
						(2) 23.3	57.2	2.1	17.4	2.45
XXIX	13.9	29.2	27.7	29.3	2.10	(1) 31.5	63.5	4.3	0.7	2.01
XXX	25.0	57.5	2.7	14.7	2.30	(1) 30.0	60.3	4.7	5.0	2.01

(1) Based on Ti by BTL (Colorimetric)

(2) Based on Ti by SUNY (X-ray fluorescence)

(3) Based on analyses by MML

TABLE II

COMPARATIVE ANALYSES OF

TITANIUM DIBORIDE (LOT V)

Lab	Titanium	Boron
A	71.54 ± 3.0	26.78
B	68.41	-
C	68.5 - 69.0	-
D	-	28.23

TABLE III

REANALYSES OF TITANIUM DIBORIDE

POWDERS (Lots XXVI & XXVII)

	Powder Lot XXVI, 840413 AFR		Powder Lot XXVII, 840416 AFR	
	+200 mesh	-200 mesh	+200 mesh	-200 mesh
Ti-Lab A	65.56	58.47	65.31	65.87
Boron				
initial	18.82	26.62	17.59	24.33
repeat	33.27	34.90	32.19	31.69
Identified* Impurities	< 2.49	< 5.22	< 1.80	< 2.33
Mass Balance				
Initial	< 86.87	< 90.31	< 84.70	< 92.53
Repeat	< 101.32	< 98.59	< 99.30	< 99.89

*Including carbon, oxygen, nitrogen and emission spectrographic analyses.

TABLE IV

SUMMARY OF CHARACTERIZATION

OF COMMERCIAL TITANIUM DIBORIDE

MATERIALS

Material Source	Reference	Analysis: Weight % Ti	Weight % B	Weight % O	Weight % C	Atom % Ti	Atom % B	Atom % O	Atom % C	B/Ti
AEE	VII - 137	63.70	-	2.40	1.56	-	-	-	-	-
CERAC	VII - 108	69.31	27.44	0.74	0.58	35.5	62.2	1.1	1.2	1.75
ESK	VIII- 15	66.56	26.32	0.77	1.65	35.5	62.1	1.2	1.2	1.75
KBI	VII - 116	69.70	30.10	0.45	0.51	33.8	64.6	0.7	1.1	1.91
KBI	VIII- 9	70.34	29.50	0.74	0.34	34.4	63.9	1.1	0.7	1.86
HCST	VI - 139	68.18	30.04	0.25	0.14	33.7	65.7	0.4	0.3	1.95
HCST	VI - 150	69.92	29.65	0.54	0.16	34.3	64.5	0.8	0.3	1.88
HCST	VII - 114	68.50	23.51	0.19	0.44	39.1	59.5	0.3	1.0	1.52
HCST	VII - 115	69.26	25.52	0.19	0.52	37.4	61.1	0.3	1.1	1.63
HCST	VIII- 20	69.86	22.65	1.59	0.15	39.8	57.2	2.7	0.3	1.44
UCC	VIII- 77	68.82	30.46	0.61	0.45	33.2	65.1	0.9	0.9	1.96

APPENDIX A

SUMMARY OF EXPERIMENTS

EXPERIMENT NO.	ART REFERENCE NUMBER	NOTEBOOK REFERENCE
I	830207	VII - 98
II	830812	- 99
III	830812 R	-100
IV	830813	-101
V	830815	-102
VI	830826-A	-103
VII	808326-B	-104
VIII	830919-A	-109
IX	830919-B	-110
X	830922-A	-111
XI	830922-B	-112
XII	831013-A	VIII- 21
XIII	831013-B	- 22
XIV	831013-C	- 23
XV	831013-D	- 24
XVI	831013-E	- 62
XVII	831013-F	- 63
XVIII	831108-A	- 64
XIX	831108-B	- 65
XX	831108-C	- 66
XXI	831108-D	- 67
XXII	831108-E	- 69
XXIII	831209	- 95
XXIV	840123 F	IX - 19
XXV	840124 F	IX - 20
XXVI	840413 AFR	X - 27
XXVII	840416 AFR	X - 27
XXVIII	840412	X - 38
XXIX	840501-P	X - 37
XXX	840412 R	X - 88

END OF PROGRAM

MIX:

TiO_2 (1):	7.25 kg	63.7 %
B_4C (2):	2.72	23.9
C (3):	1.41	12.4

COMPOSITION:

	kg	moles	mole %
Ti:	4.38	91.4	14.6
B:	2.10	194.6	31.0
O:	2.68	178.5	28.4
C:	1.96	163.2	26.0

FIRING:

Temp Range: 2050 to 2100 °C

Weight of Feed: 9.1 kg.

Feed Rate: 11.4 kg/hr.

Weight of Product: n.a. (4) kg.

ANALYSIS:

Analytical Number: 480,617

Ti (BTL, Colorimetric)	69.10	%
(SUNY, XRF)	n.a.	%
B	28.30	%
C	4.66	%
O	0.36	%
N	0.045	%

Spectrographic

Ca	1 - 5	%
Mo	0.2	%
Fe	0.1	%
others	< 0.006	%

X-RAY DIFFRACTION:

$\frac{I\ (2\theta \approx 44.5°)}{I\ (2\theta \approx 42°)}$ — 77 / 2

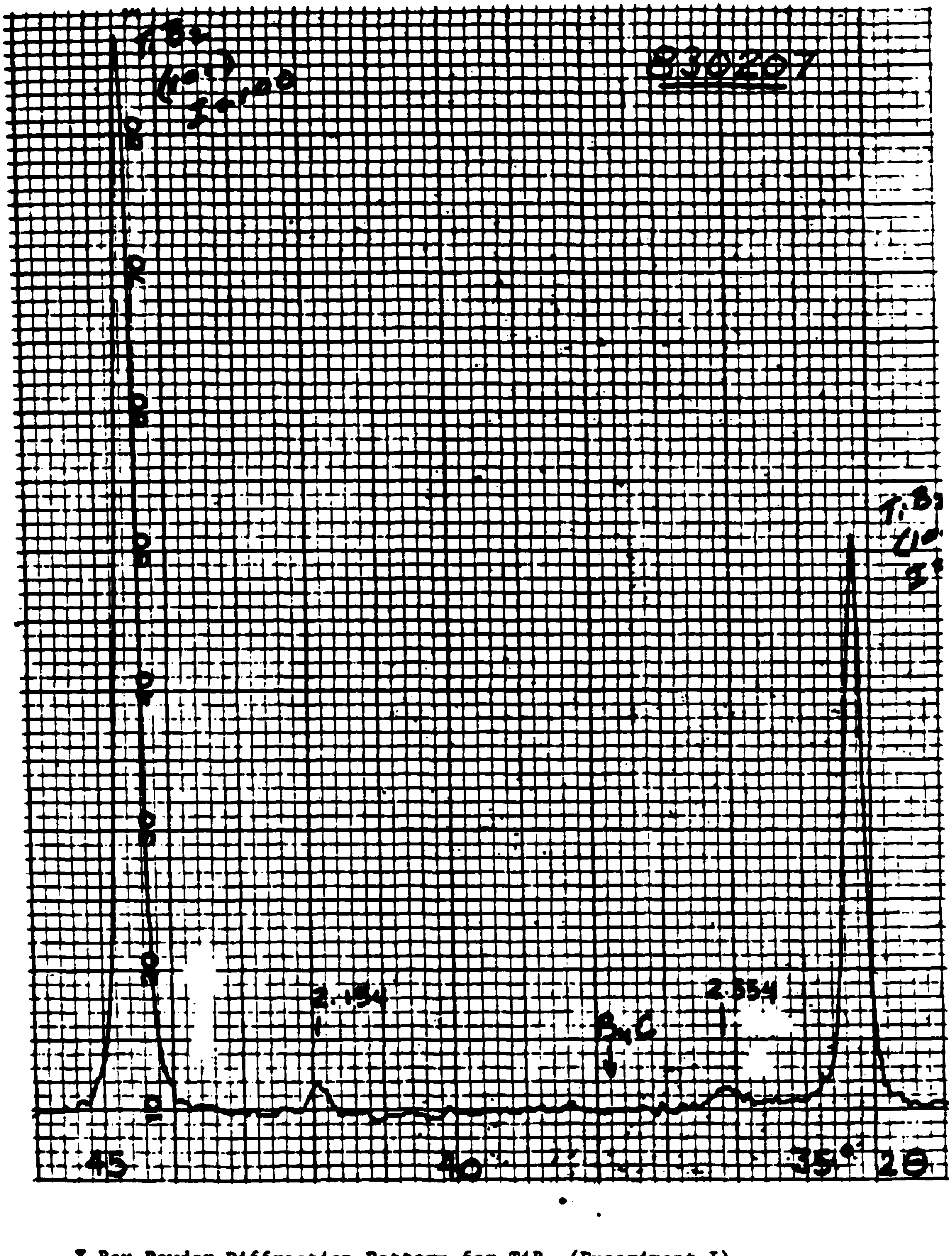
SURFACE AREA:

n.a. m^2/g

PARTICLE SIZE (MICROTRAC):

MV	n.a.	µm
PH	n.a.	µm
PM	n.a.	µm
PS	n.a.	µm

( ) See list of footnotes at end of Appendix A



X-Ray Powder Diffraction Pattern for TiB_2 (Experiment I)

EXPT. NO. II

ART REF. NO. 830812

MIX:

TiO_2 (1):	7.25 kg	63.7 %
B_4C (5):	2.72	23.9
C (3):	1.41	12.4

COMPOSITION:

Ti:	4.38 kg	91.4 moles	14.5 mole %
B :	1.96	181.2	28.7
O :	2.91	181.9	28.8
C :	2.12	176.3	27.9

FIRING:

Temp Range: 2020 to 2120 °C

Weight of Feed: 36.3 kg.

Feed Rate: 16.8 kg/hr.

Weight of Product: n.a. kg.

ANALYSIS:

Analytical Number: 968

Ti(BTL, Colorimetric)	63.15	%
(SUNY, XRF)	67.25	%
B	24.94	%
C	4.63	%
O	3.79	%
N	0.46	%
Spectrographic		
Ca	0.2	%
Fe, Si, Zr, Mg	0.01	%
		%
Others	< 0.01	%

X-RAY DIFFRACTION:

$$\frac{I\ (2\theta \approx 44.5°)}{I\ (2\theta \approx 42°)} = \frac{54}{4.5}$$

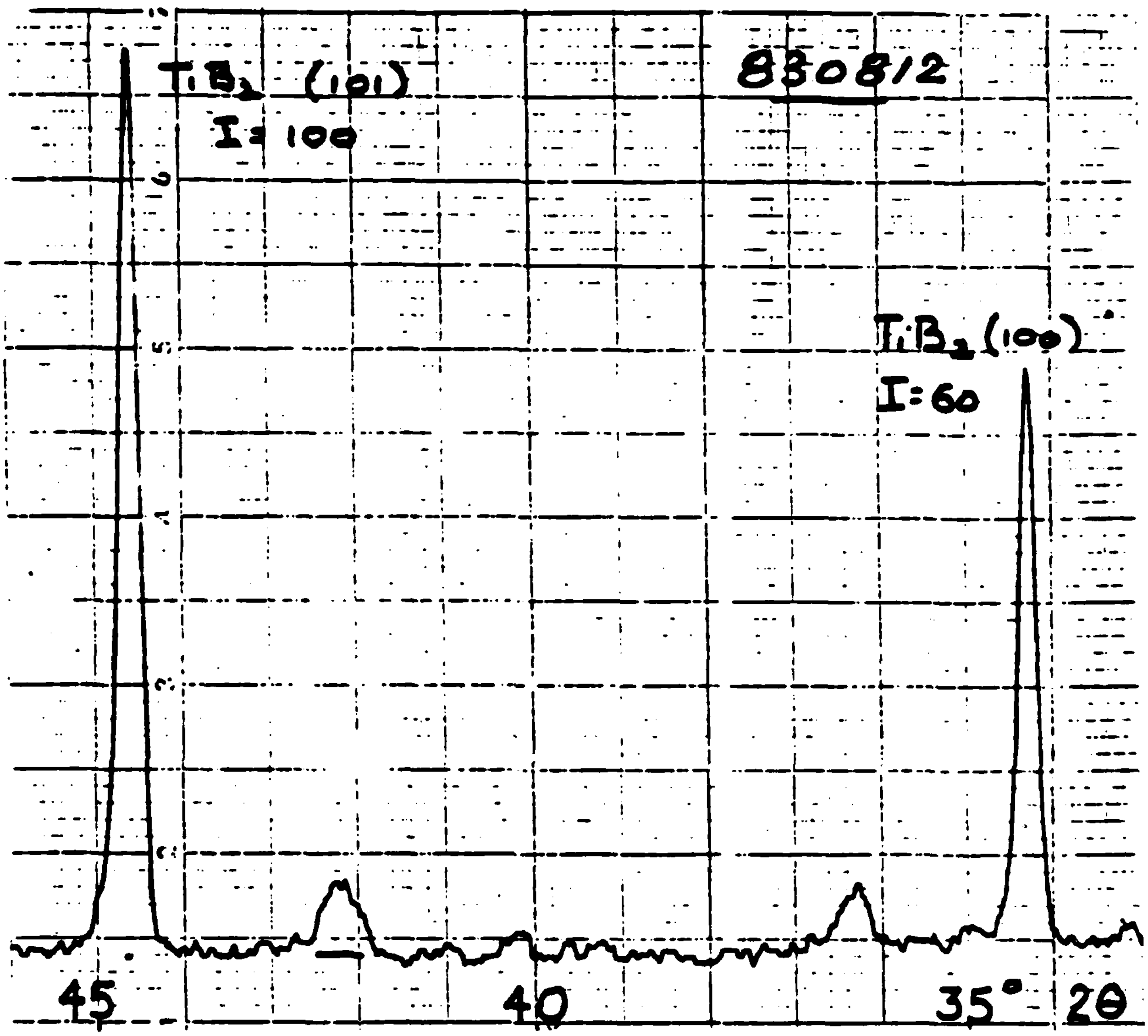
SURFACE AREA:

0.98 m^2/g

PARTICLE SIZE (MICROTRAC):

MV	n.a.	µm
PH	n.a.	µm
PM	n.a.	µm
PS	n.a.	µm

( ) See list of footnotes at end of Appendix A.



X-Ray Powder Diffraction Pattern for TiB_2 (Experiment II)

MIX:

TiO_2( ): (6) kg ____ %

B_4C ( ): ____ ____

C ( ): ____ ____

COMPOSITION:

	kg	moles	mole %
Ti:			
B :			
O :			
C :			

FIRING:

Temp Range: 2095 to 2145 °C

Weight of Feed: n.a. kg.

Feed Rate: n.a. kg/hr.

Weight of Product: n.a. kg.

ANALYSIS:

Analytical Number: 968

Ti (BTL, Colorimetric)	67.85	%
(SUNY, XRF)	68.3	%
B	27.65	%
C	1.48	%
O	1.12	%
N	0.37	%
Spectrographic		
Ca	0.1	%
Fe	0.06	%
Si, W	0.02	%
Others	< 0.01	%

X-RAY DIFFRACTION:

$$\frac{I\ (2\theta \cong 44.5°)}{I\ (2\theta \cong 42°)} \qquad \frac{62}{4}$$

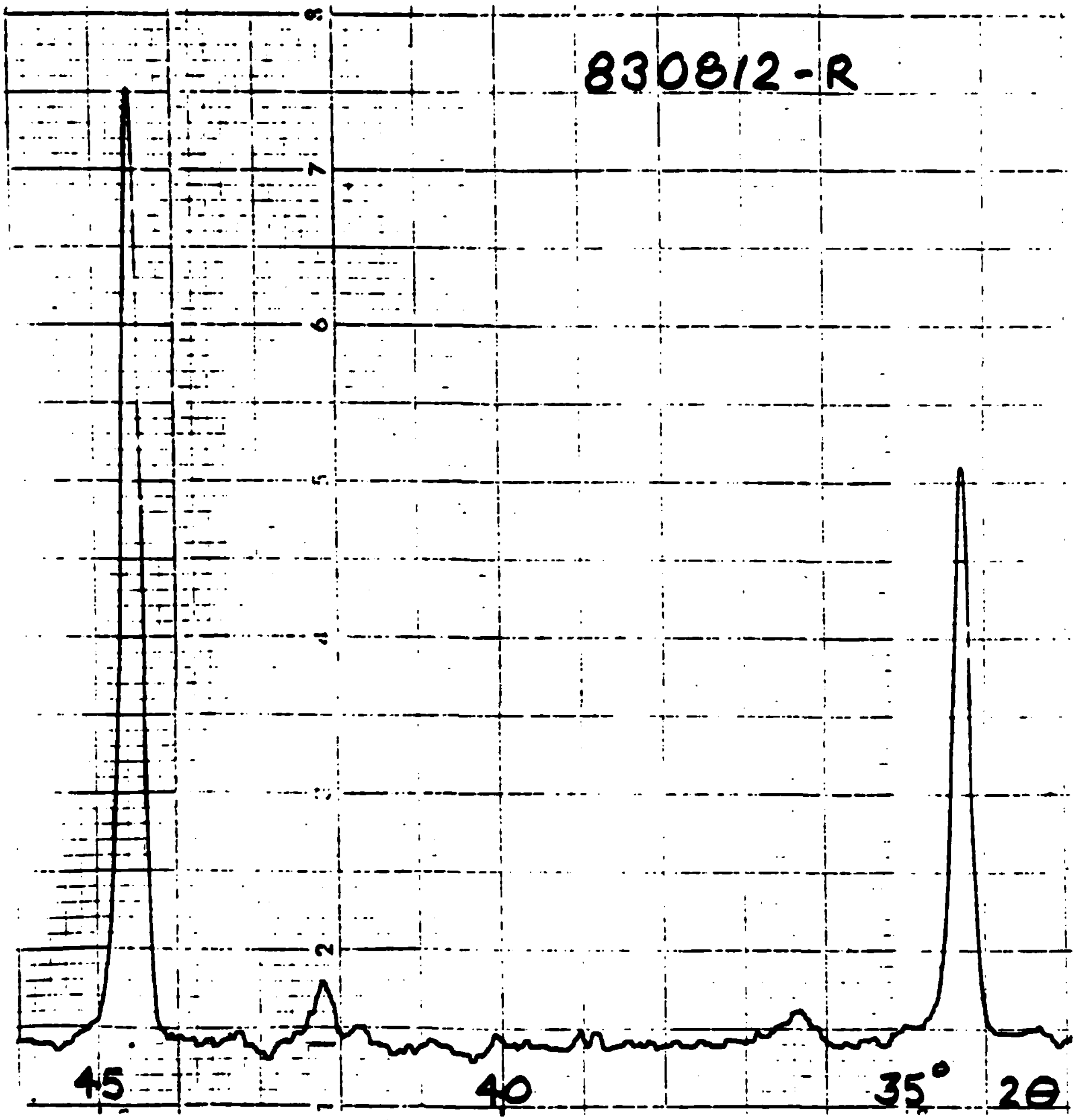
SURFACE AREA:

0.61 m^2/g

PARTICLE SIZE (MICROTRAC):

MV	n.a.	µm
PH	n.a.	µm
PM	n.a.	µm
PS	n.a.	µm

( ) See list of footnotes at end of Appendix A.



X-Ray Powder Diffraction Pattern for TiB_2 (Experiment III)

MIX:

TiO_2 (1):	7.25 kg	63.7 %
B_4C (5):	2.72	23.9
C (3):	1.41	12.4

COMPOSITION:

	kg	moles	mole %
Ti:	4.38	91.4	14.5
B :	1.96	181.3	28.7
O :	2.91	181.9	28.8
C :	2.12	176.3	27.9

FIRING:

Temp Range: 2050 to 2120 °C

Weight of Feed: n.a. kg.

Feed Rate: n.a. kg/hr.

Weight of Product: n.a. kg.

ANALYSIS:

Analytical Number: 968

Ti (BTL, Colorimetric)	65.76	%
(SUNY, XRF)	68.3	%
B	27.84	%
C	1.03	%
O	1.83	%
N	0.27	%
Spectrographic		
Ca	0.1	%
Fe	0.06	%
Si	0.02	%
Others	< 0.01	%

X-RAY DIFFRACTION:

$$\frac{I\ (2\theta \approx 44.5°)}{I\ (2\theta \approx 42°)} \quad \frac{65}{1}$$

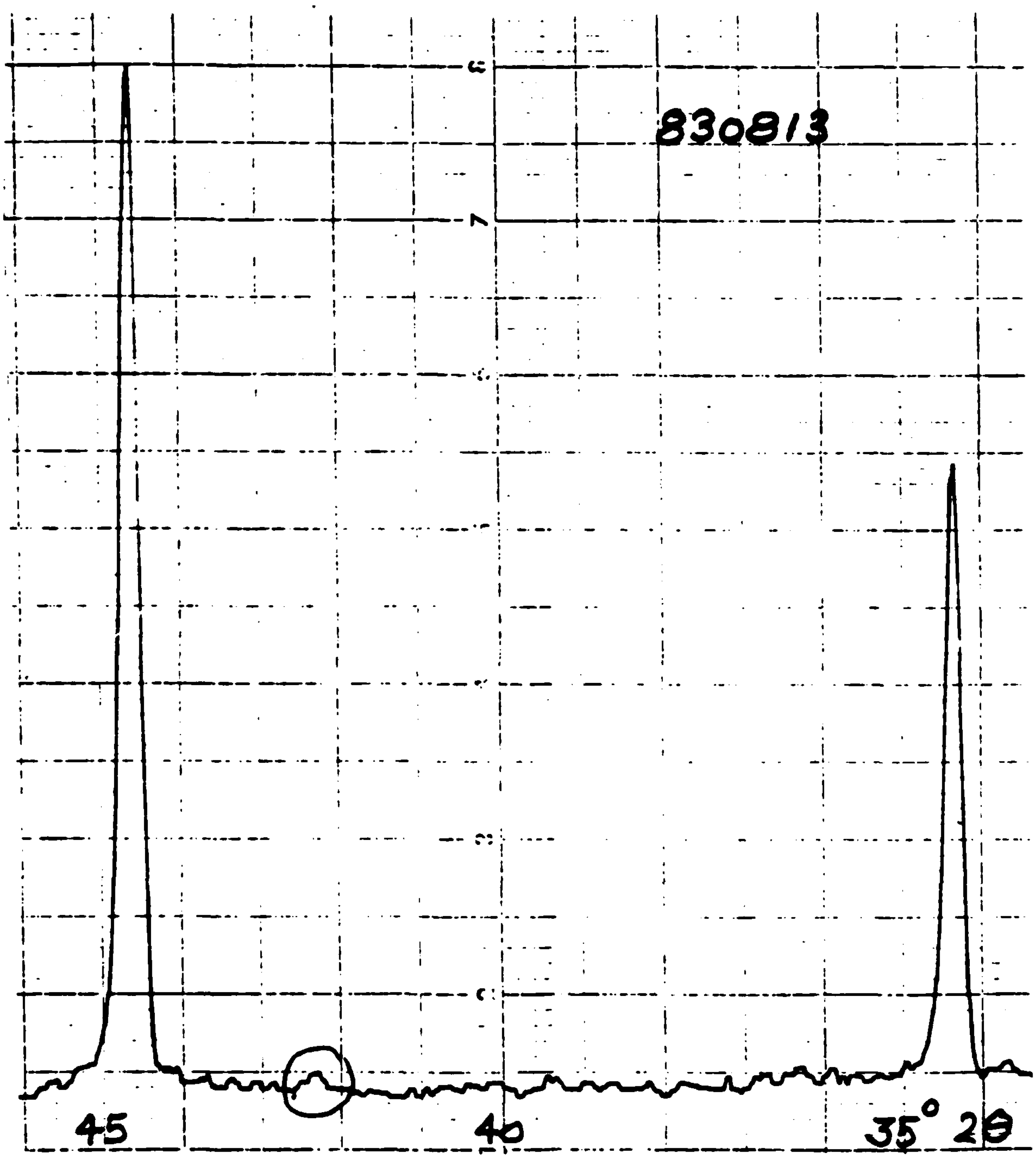
SURFACE AREA:

0.44 m^2/g

PARTICLE SIZE (MICROTRAC):

MV	n.a.	μm
PH	n.a.	μm
PM	n.a.	μm
PS	n.a.	μm

( ) See list of footnotes at end of Appendix A.



X-Ray Powder Diffraction Pattern for TiB_2 (Experiment IV)

MIX:

TiO_2 (1):	7.25 kg	63.7 %
B_4C (5):	2.72	23.9
C (3):	1.41	12.4

COMPOSITION:

	kg	moles	mole %
Ti:	4.38	91.4	14.5
B :	1.96	181.3	28.7
O :	2.91	181.9	28.8
C :	2.12	176.3	27.9

FIRING:

Temp Range: 1970 to 2020 °C

Weight of Feed: n.a. kg.

Feed Rate: n.a. kg/hr.

Weight of Product: n.a. kg.

ANALYSIS:

Analytical Number: 968, 1021

Ti (17) (BTL, Colorimetric)	68.41	%
(SUNY, XRF)	68.75	%
B (17)	28.23	%
C	0.93	%
O	1.17	%
N	0.35	%
Spectrographic		
Ca	0.2	%
Fe	0.1	%
		%
Others	< 0.01	%

X-RAY DIFFRACTION:

$\frac{I\ (2\theta \approx 44.5°)}{I\ (2\theta \approx 42°)}$ 68 / 1.5

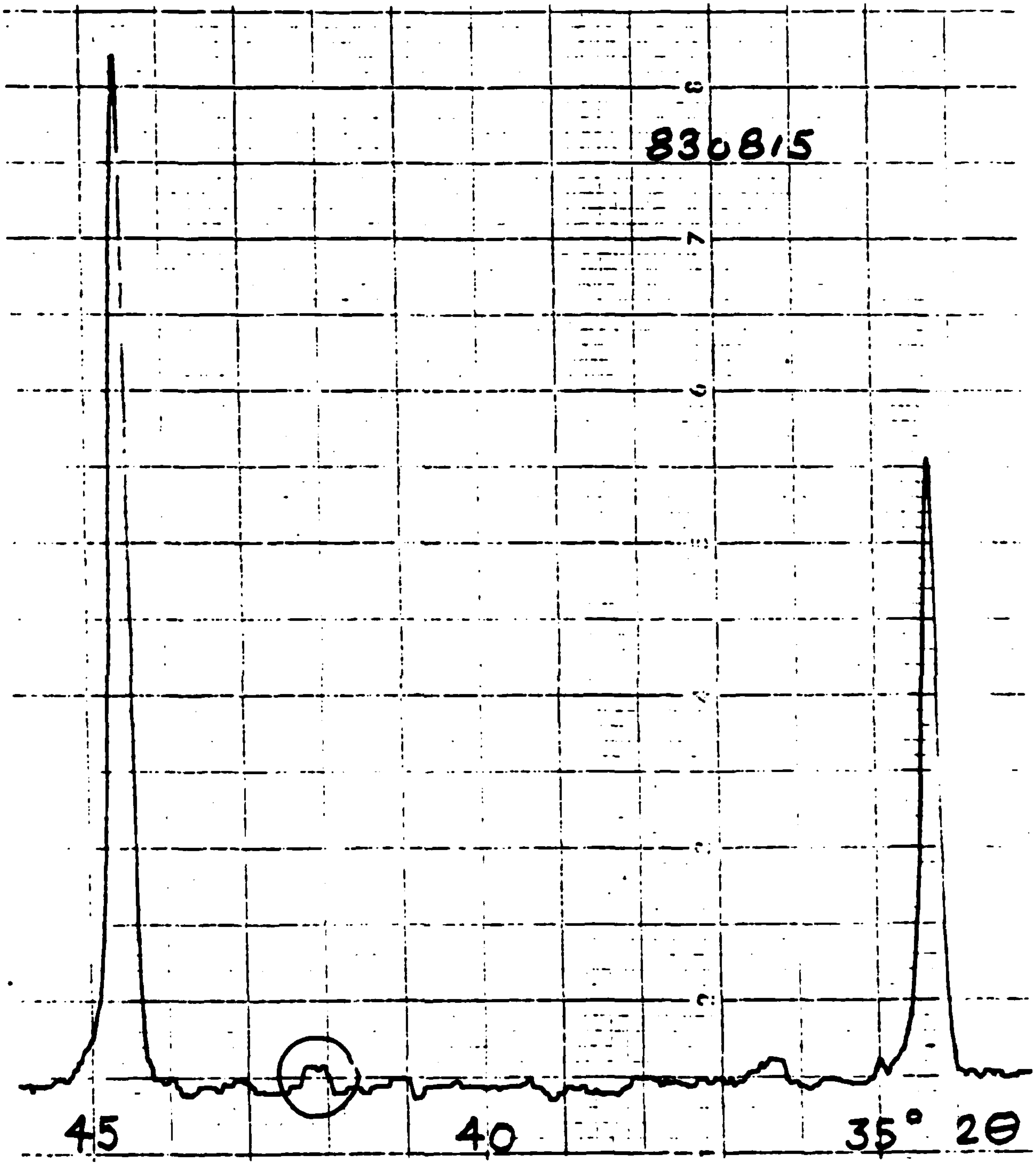
SURFACE AREA:

0.63 m^2/g

PARTICLE SIZE (MICROTRAC):

MV	13	μm
PH	25	μm
PM	10	μm
PS	4.3	μm

( ) See list of footnotes at end of Appendix A.



X-Ray Powder Diffraction Pattern for TiB_2 (Experiment V)

EXPT. NO. VI

ART REF. NO. 830826-A

MIX:

TiO_2 (1):	29.06 kg	63.4 %
B_4C (7):	11.35	24.8
C (3):	5.45	11.9

COMPOSITION:

	kg	moles	mole %
Ti:	17.55	366.4	14.6
B :	8.27	765.4	30.6
O :	11.68	729.9	29.1
C :	7.73	643.6	25.7

FIRING:

Temp Range: 1950 to 2030 °C

Weight of Feed: 22.7 kg.

Feed Rate: n.a. kg/hr.

Weight of Product: 9.5 kg.

ANALYSIS:

Analytical Number: 968

Ti (BTL, Colorimetric)	65.83	%
(SUNY, XRF)	68.3	%
B	27.85	%
C	0.75	%
O	1.53	%
N	0.44	%
Spectrographic		
Ca	0.2	%
Fe	0.1	%
Si	0.02	%
others	< 0.01	%

X-RAY DIFFRACTION:

$\frac{I\ (2\theta \cong 44.5^\circ)}{I\ (2\theta \cong 42^\circ)}$ $\frac{63}{2}$

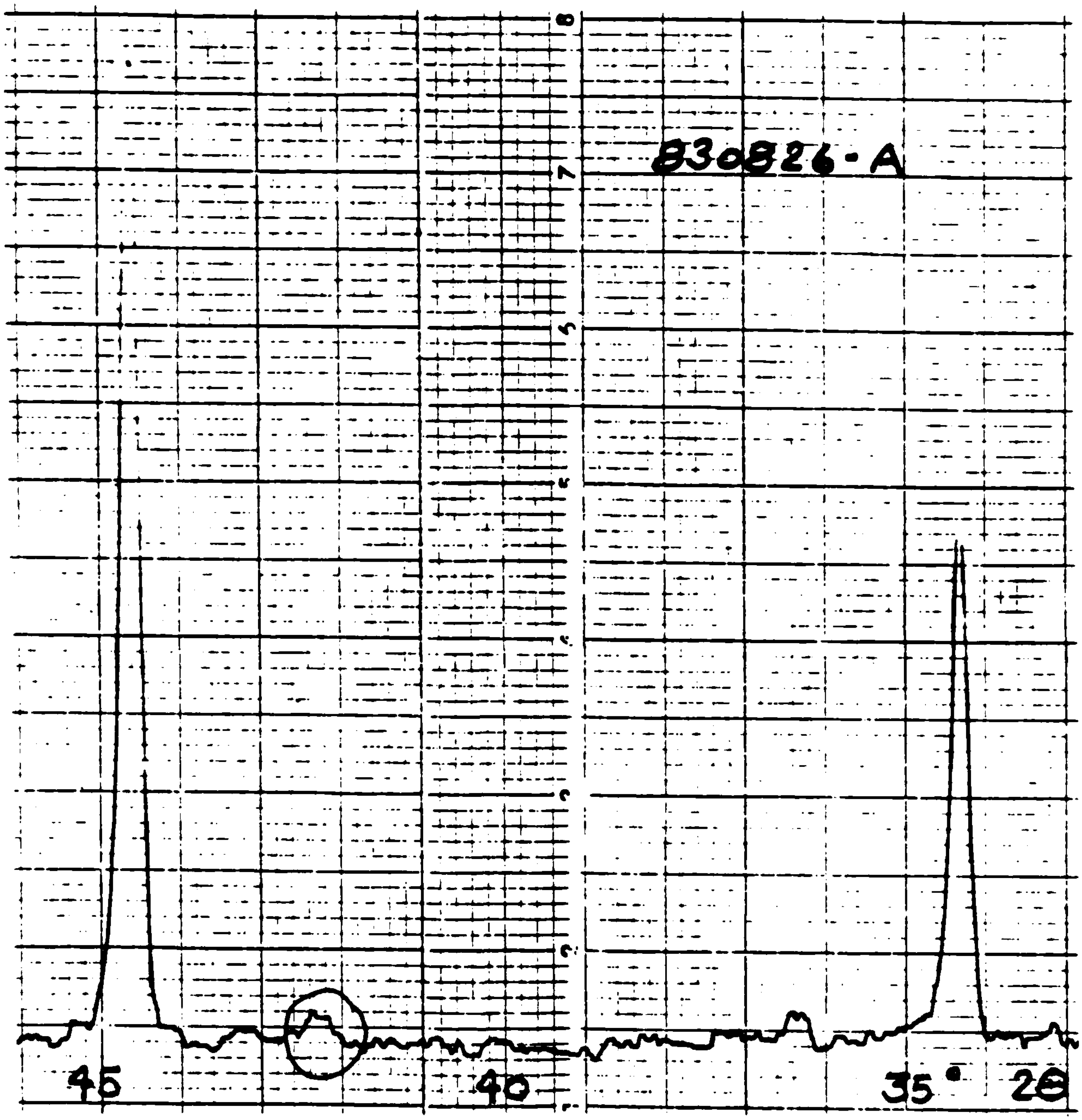
SURFACE AREA:

0.59 m^2/g

PARTICLE SIZE (MICROTRAC):

MV	n.a.	μm
PH	n.a.	μm
PM	n.a.	μm
PS	n.a.	μm

( ) See list of footnotes at end of Appendix A.



X-Ray Powder Diffraction Pattern for TiB_2 (Experiment VI)

EXPT. NO. VII

ART REF. NO. 830826-B

MIX:

TiO_2 (1):	29.06 kg	63.4 %
B_4C (7):	11.35	24.8
C (3):	5.45	11.9

COMPOSITION:

	kg	moles	mole %
Ti:	17.55	366.4	14.6
B:	8.27	765.4	30.6
O:	11.68	729.9	29.1
C:	7.73	643.6	25.7

FIRING:

Temp Range: 2100 to 2130 °C

Weight of Feed: 22.7 kg.

Feed Rate: 9.1 kg/hr.

Weight of Product: 14.5 kg.

ANALYSIS:

Analytical Number: 968

Ti (BTL, Colorimetric)	67.72	%
(SUNY, XRF)	68.1	%
B	28.03	%
C	1.20	%
O	0.57	%
N	0.17	%
Spectrographic		
Ca, Fe	0.1	%
Si	0.02	%
		%
Others	< 0.01	%

X-RAY DIFFRACTION:

$$\frac{I\ (2\theta \cong 44.5°)}{I\ (2\theta \cong 42°)} \quad \frac{69}{<1}$$

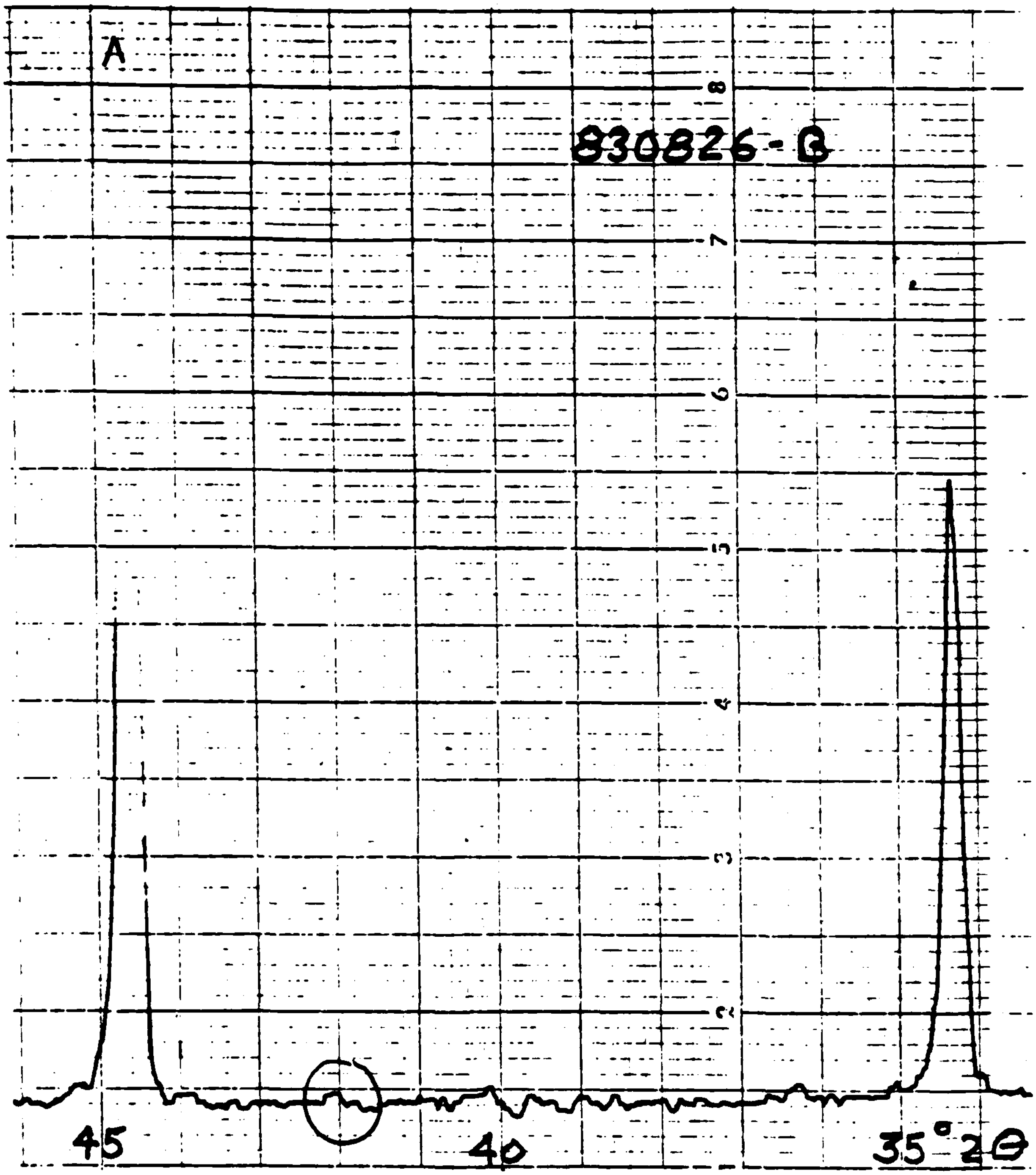
SURFACE AREA:

0.42 m^2/g

PARTICLE SIZE (MICROTRAC):

MV	n.a.	μm
PH	n.a.	μm
PM	n.a.	μm
PS	n.a.	μm

( ) See list of footnotes at end of Appendix A.



X-Ray Powder Diffraction Pattern for TiB_2 (Experiment VII)

EXPT. NO. VIII

ART REF. NO. 830919-A

MIX:

TiO_2 (1):	29.51 kg	65.0 %
B_4C (7):	11.35	25.0
C (3):	4.54	10.0

COMPOSITION:

	kg	moles	mole %
Ti:	17.82	372.0	15.2
B :	8.27	765.4	31.3
O :	11.86	741.0	30.3
C :	6.82	567.9	23.2

FIRING:

Temp Range: 1990 to 2060 °C

Weight of Feed: 21.8 kg.

Feed Rate: n.a. kg/hr.

Weight of Product: 10.4 kg.

ANALYSIS:

Analytical Number: 1027

Ti (BTL, Colorimetric)	69.35	%
(SUNY, XRF)	70.6	%
B	27.2	%
C	1.39	%
O	0.41	%
N	0.42	%
Spectrographic		
Ca	0.4	%
Fe	0.1	%
Si	0.06	%
Others	< 0.002	%

X-RAY DIFFRACTION:

$\dfrac{I\ (2\theta \approx 44.5°)}{I\ (2\theta \approx 42°)}$ 58 / 2.5

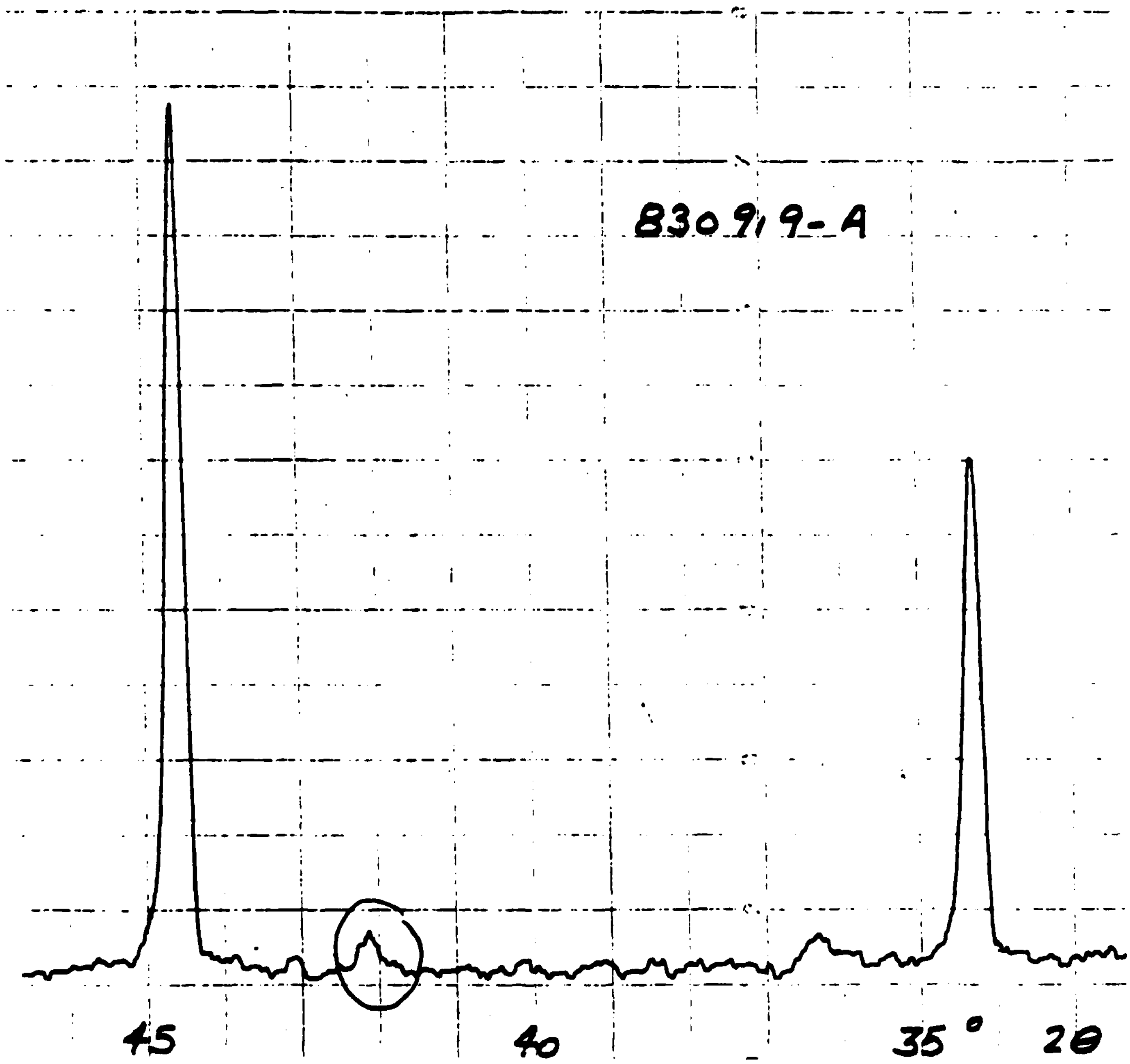
SURFACE AREA:

0.32 m^2/g

PARTICLE SIZE (MICROTRAC):

MV	28	μm
PH	82	μm
PM	12	μm
PS	4	μm

( ) See list of footnotes at end of Appendix A.



X-Ray Powder Diffraction Pattern for TiB_2 (Experiment VIII)

EXPT. NO. IX

ART REF. NO. 830919-B

MIX:

TiO_2 (1):	29.51 kg	65.0 %	
B_4C (7):	11.35	25.0	
C (3):	4.54	10.0	

COMPOSITION:

	kg	moles	mole %
Ti:	17.82	372.0	15.2
B :	8.27	765.4	31.3
O :	11.86	741.0	30.3
C :	6.82	567.9	23.2

FIRING:

Temp Range: 2085 to 2110 °C

Weight of Feed: 21.8 kg.

Feed Rate: n.a. kg/hr.

Weight of Product: 10.4 kg.

ANALYSIS:

Analytical Number: 1027

Ti (BTL, Colorimetric)	69.78	%
(SUNY, XRF)	69.8	%
B	26.03	%
C	0.97	%
O	0.28	%
N	0.31	%
Spectrographic		
Ca	0.4	%
Fe	0.06	%
Si	0.02	%
Others	< 0.002	%

X-RAY DIFFRACTION:

$$\frac{I\ (2\theta \cong 44.5^\circ)}{I\ (2\theta \cong 42^\circ)} \qquad \frac{64}{3}$$

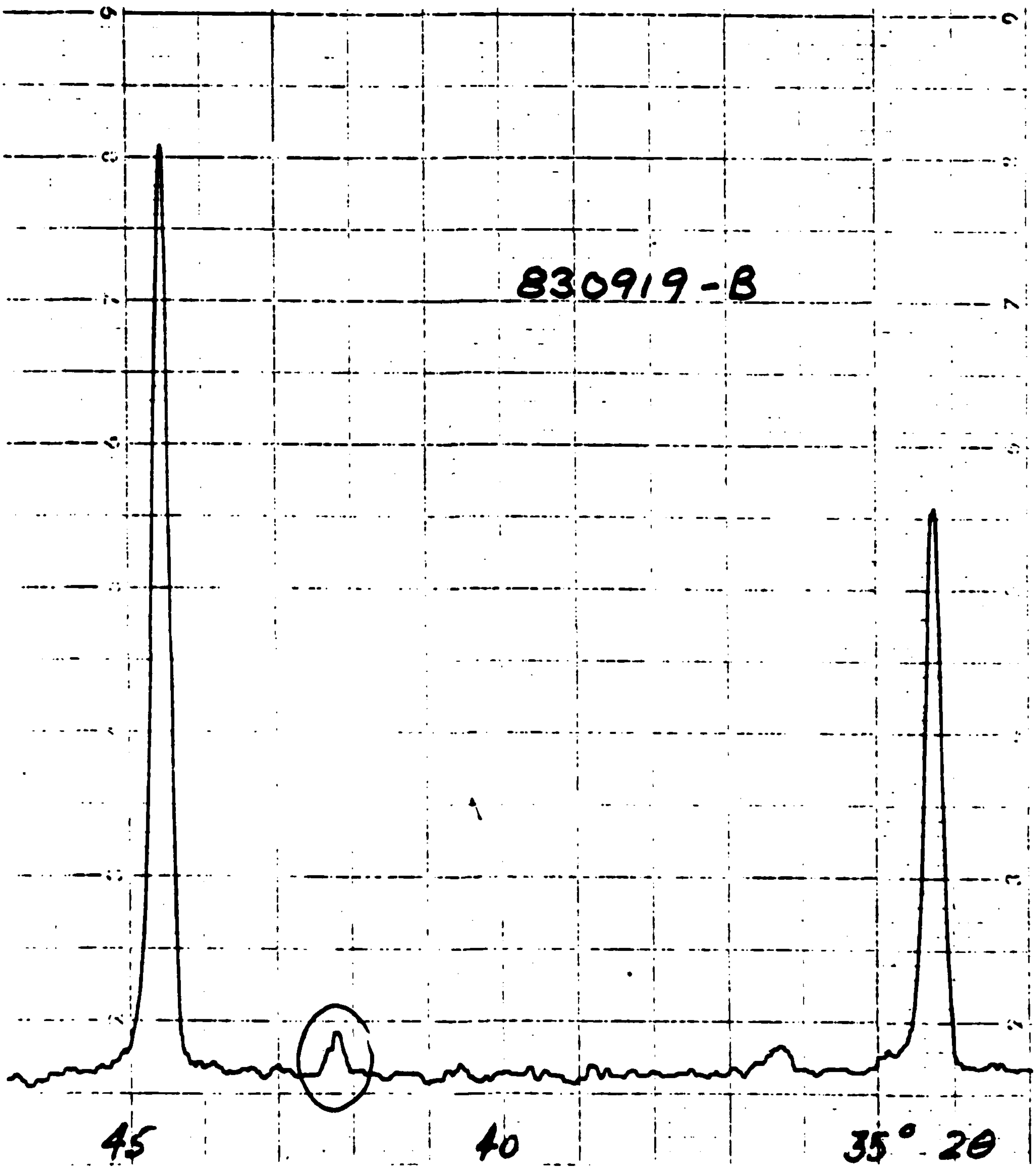
SURFACE AREA:

0.43 m^2/g

PARTICLE SIZE (MICROTRAC):

MV	29	μm
PH	79	μm
PM	14	μm
PS	4	μm

( ) See list of footnotes at end of Appendix A.



X-Ray Powder Diffraction Pattern for TiB_2 (Experiment IX)

EXPT. NO. X

ART REF. NO. 830922-A

MIX:

TiO_2 (1):	29.06	kg	64.0	%
B_4C (7):	11.80		26.0	
C (3):	4.54		10.0	

COMPOSITION:

Ti:	17.55	kg	366.4	moles	14.8	mole %
B :	8.60		795.8		32.2	
O :	11.69		730.5		29.6	
C :	6.91		575.4		23.3	

FIRING:

Temp Range: 1980 to 2060 °C

Weight of Feed: 22.0 kg.

Feed Rate: n.a. kg/hr.

Weight of Product: 10.4 kg.

ANALYSIS:

Analytical Number: 1027

Ti (BTL, Colorimetric)	67.01	%
(SUNY, XRF)	70.1	%
B	24.87	%
C	2.24	%
O	0.56	%
N	0.25	%
Spectrographic		
Ca	0.6	%
Si	0.2	%
Fe	0.1	%
Others	< 0.004	%

X-RAY DIFFRACTION:

$$\frac{I\ (2\theta \cong 44.5°)}{I\ (2\theta \cong 42°)} \qquad \frac{70}{3}$$

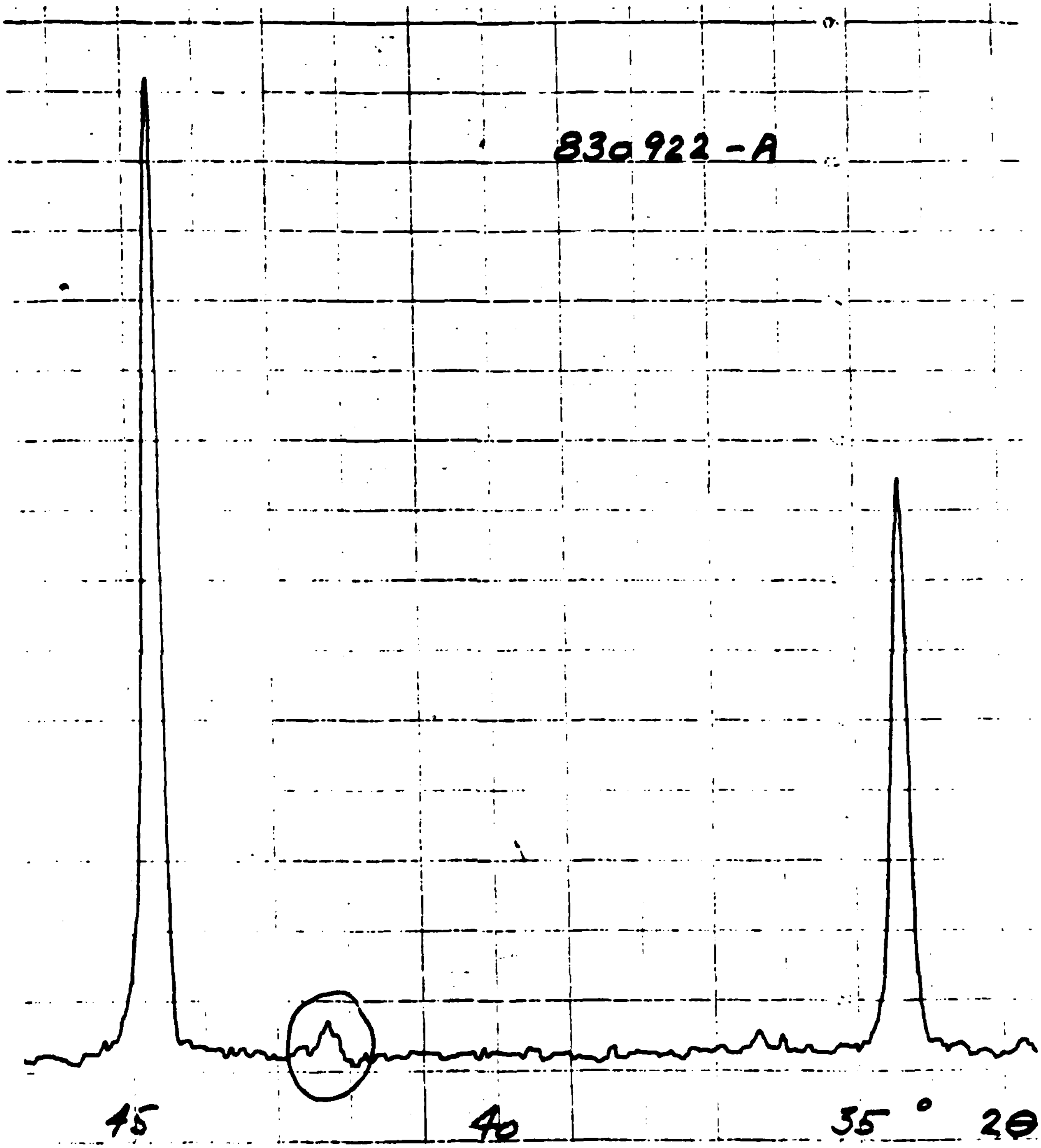
SURFACE AREA:

0.63 m^2/g

PARTICLE SIZE (MICROTRAC):

MV	37	μm
PH	104	μm
PM	16	μm
PS	5	μm

( ) See list of footnotes at end of Appendix A.



X-Ray Powder Diffraction Pattern for TiB_2 (Experiment X)

EXPT. NO. XI

ART REF. NO. 830922-B

MIX:

TiO_2 (1):	29.06 kg	64.0 %
B_4C (7):	11.80	26.0
C (3):	4.54	10.0

COMPOSITION:

	kg	moles	mole %
Ti:	17.55	366.4	14.8
B:	8.60	795.8	32.2
O:	11.69	730.5	29.6
C:	6.91	575.4	23.3

FIRING:

Temp Range: 2040 to 2080 °C

Weight of Feed: 22.7 kg.

Feed Rate: n.a. kg/hr.

Weight of Product: 10.4 kg.

ANALYSIS:

Analytical Number: 1027

Ti (BTL, Colorimetric)	68.28 %
(SUNY, XRF)	69.21 %
B	26.39 %
C	1.52 %
O	0.50 %
N	0.41 %
Spectrographic	
Ca	0.4 %
Si	0.2 %
Fe	0.1 %
Others	< 0.002 %

X-RAY DIFFRACTION:

$$\frac{I\ (2\theta \approx 44.5°)}{I\ (2\theta \approx 42°)} \qquad \frac{65}{2}$$

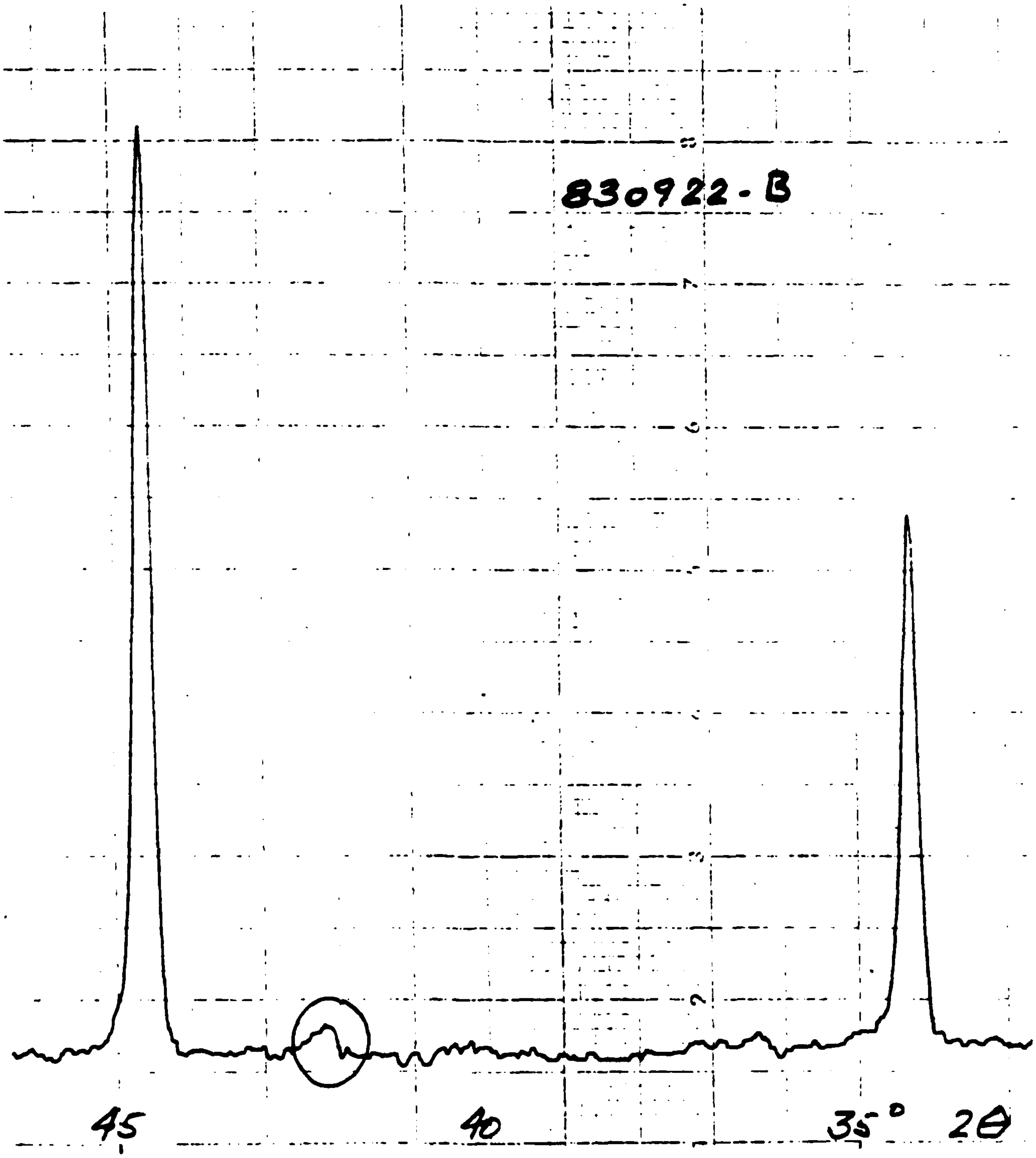
SURFACE AREA:

0.56 m^2/g

PARTICLE SIZE (MICROTRAC):

MV	29 μm
PH	76 μm
PM	15 μm
PS	5 μm

( ) See list of footnotes at end of Appendix A.



X-Ray Powder Diffraction Pattern for TiB_2 (Experiment XI)

EXPT. NO. XII

ART REF. NO. 831013-A

MIX:

TiO_2 (1):	22.70 kg	63.5 %
B_4C (8):	8.27	23.1
C (3):	4.79	13.4

COMPOSITION:

	kg	moles	mole %
Ti:	13.75	287.1	14.5
B :	6.21	574.5	29.0
O :	8.91	557.3	28.2
C :	6.73	560.7	28.3

FIRING:

Temp Range: 2000 to 2000 °C

Weight of Feed: 18.6 kg.

Feed Rate: 8.2 kg/hr.

Weight of Product: 7.7 kg.

ANALYSIS:

Analytical Number: 1135

Ti (BTL, Colorimetric)	66.25	%
(SUNY, XRF)	70.7	%
B	24.74	%
C	3.85	%
O	0.72	%
N	0.16	%

Spectrographic

Ca	0.2	%
Fe	0.08	%
Si	0.06	%
Others	< 0.05	%

X-RAY DIFFRACTION:

$$\frac{I\ (2\theta \cong 44.5°)}{I\ (2\theta \cong 42°)} \qquad \frac{64}{4}$$

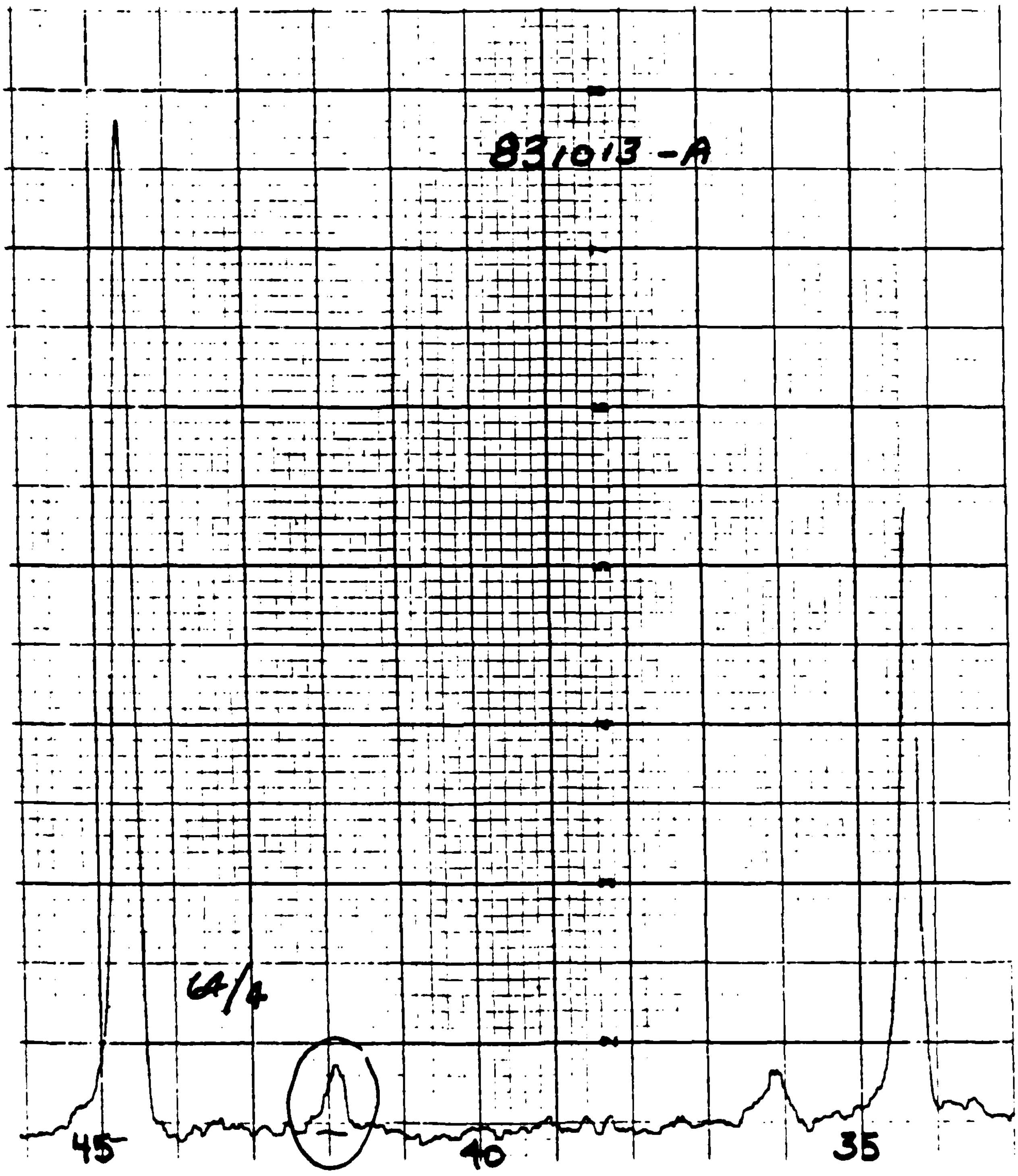
SURFACE AREA:

0.64 m^2/g

PARTICLE SIZE (MICROTRAC):

MV	22	μm
PH	40	μm
PM	17	μm
PS	6.5	μm

( ) See list of footnotes at end of Appendix A.



X-Ray Powder Diffraction Pattern for TiB_2 (Experiment XII)

EXPT. NO. XIII

ART REF. NO. 831013-B

MIX:

TiO_2 (1):	22.70	kg	63.5	%
B_4C (8):	8.27		23.1	
C (3):	4.79		13.4	

COMPOSITION:

Ti:	13.75	kg	287.1	moles	14.5	mole %
B :	6.21		574.5		29.0	
O :	8.91		557.3		28.2	
C :	6.73		560.7		28.3	

FIRING:

Temp Range: 2095 to 2120 °C

Weight of Feed: 19.0 kg.

Feed Rate: n.a. kg/hr.

Weight of Product: 7.7 kg.

ANALYSIS:

Analytical Number: 1135

Ti (BTL, Colorimetric)	62.90	%
(SUNY, XRF)	69.1	%
B	21.59	%
C	3.51	%
O	1.51	%
N	0.32	%
Spectrographic		
Fe	0.3	%
Ca	0.1	%
Si	0.08	%
Others	< 0.05	%

X-RAY DIFFRACTION:

$$\frac{I\ (2\theta \cong 44.5°)}{I\ (2\theta \cong 42°)} = \frac{70}{4}$$

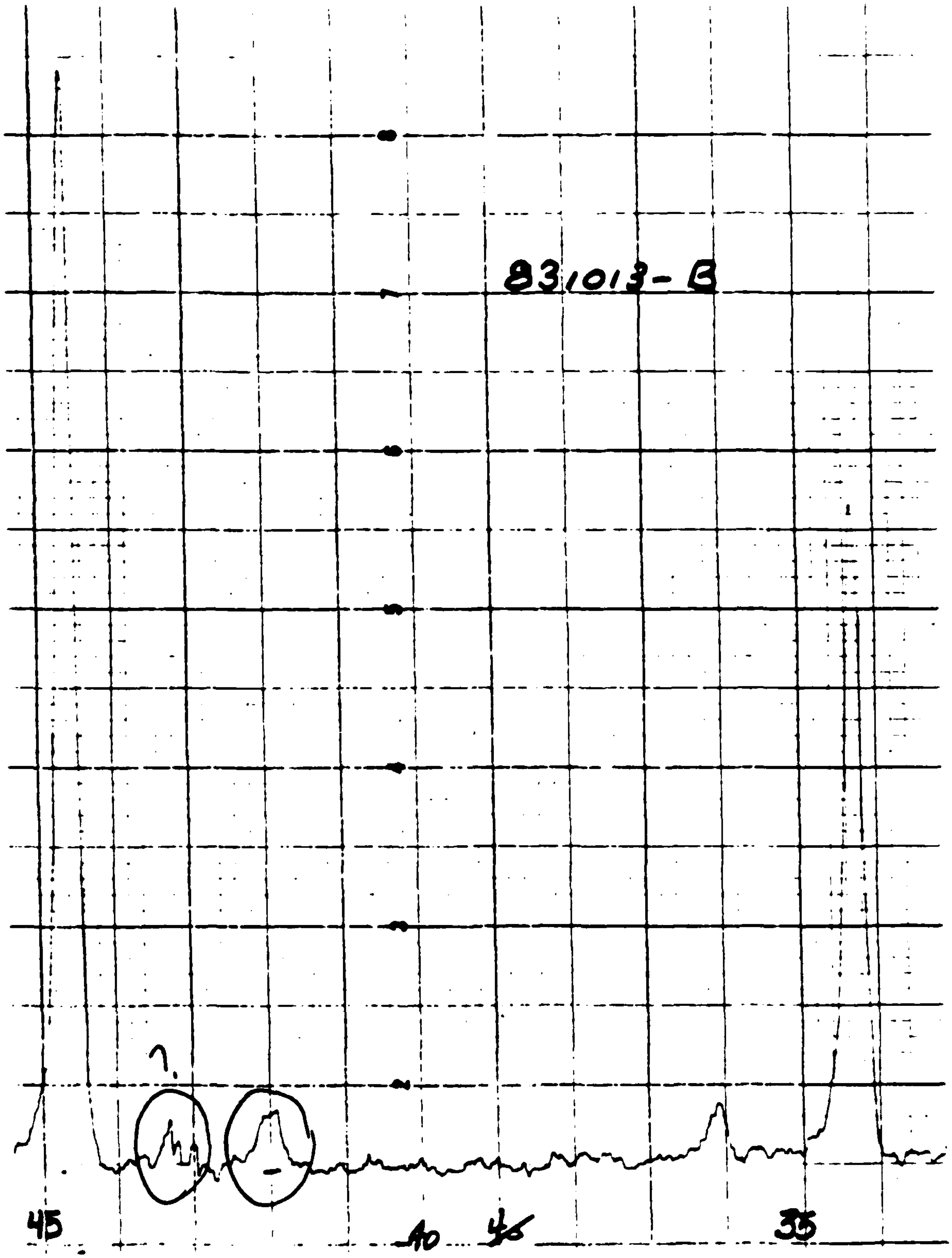
SURFACE AREA:

0.55 m^2/g

PARTICLE SIZE (MICROTRAC):

MV	21	μm
PH	37	μm
PM	16	μm
PS	6	μm

( ) See list of footnotes at end of Appendix A.



X-Ray Powder Diffraction Pattern for TiB_2 (Experiment XIII)

MIX:

TiO_2 (1):	22.70	kg	63.8	%
B_4C (8):	8.26		23.2	
C (3):	4.65		13.0	

COMPOSITION:

Ti:	13.75	kg	287.0	moles	14.6 mole %
B :	6.21		574.5		29.2
O :	8.94		558.8		28.4
C :	6.59		548.7		27.9

FIRING:

Temp Range: 1995 to 2010 °C

Weight of Feed: 15.0 kg.

Feed Rate: 10.0 kg/hr.

Weight of Product: 8.2 kg.

ANALYSIS:

Analytical Number: 1135

Ti (BTL, Colorimetric)	64.22	%
(SUNY, XRF)	72.3	%
B	27.54	%
C	1.97	%
O	0.42	%
N	0.14	%
Spectrographic		
Fe	0.2	%
Ca	0.1	%
Si	0.08	%
Others	< 0.05	%

X-RAY DIFFRACTION:

$$\frac{I\ (2\theta \cong 44.5°)}{I\ (2\theta \cong 42°)} \qquad \frac{50}{1}$$

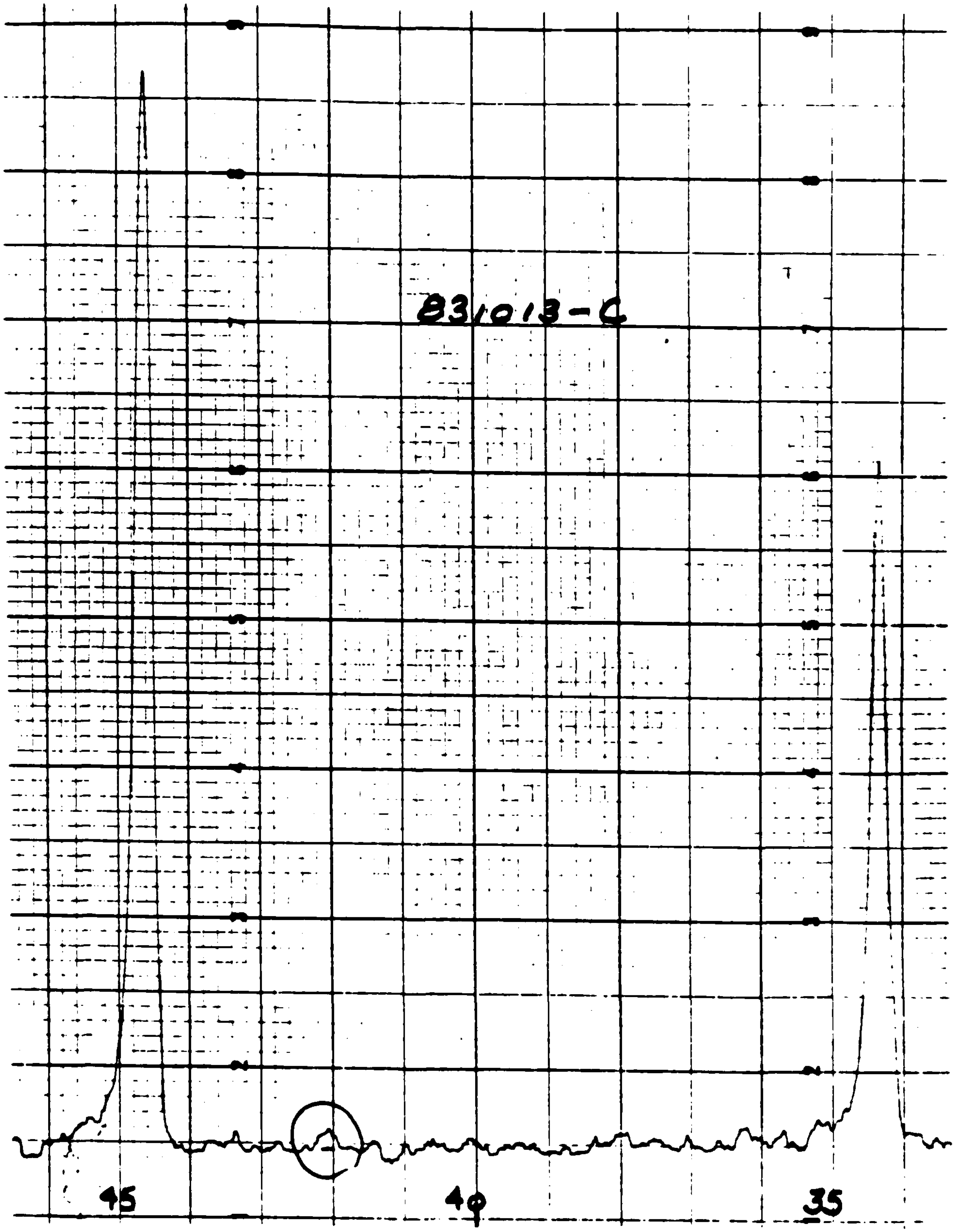
SURFACE AREA:

0.67 m^2/g

PARTICLE SIZE (MICROTRAC):

MV	17	µm
PH	29	µm
PM	15	µm
PS	6.0	µm

( ) See list of footnotes at end of Appendix A.



X-Ray Powder Diffraction Pattern for TiB_2 (Experiment XIV)

MIX:

TiO_2 (1):	22.70 kg	63.8 %
B_4C (8):	8.26	23.2
C (3):	4.65	13.0

COMPOSITION:

Ti:	13.75 kg	287.0 moles	14.6 mole %
B :	6.21	574.5	29.2
O :	8.94	558.8	28.4
C :	6.59	548.7	27.9

FIRING:

Temp Range: 2105 to 2120 °C

Weight of Feed: 15.0 kg.

Feed Rate: 10.0 kg/hr.

Weight of Product: 8.2 kg.

ANALYSIS:

Analytical Number: 1135

Ti (BTL, Colorimetric)	66.34 %
(SUNY, XRF)	71.1 %
B	25.03 %
C	1.74 %
O	0.40 %
N	0.07 %
Spectrographic	
Fe	0.3 %
Ca	0.2 %
Si	0.06 %
Others	< 0.05 %

X-RAY DIFFRACTION:

I (2θ ≅ 44.5°)	69
I (2θ ≅ 42°)	0.5

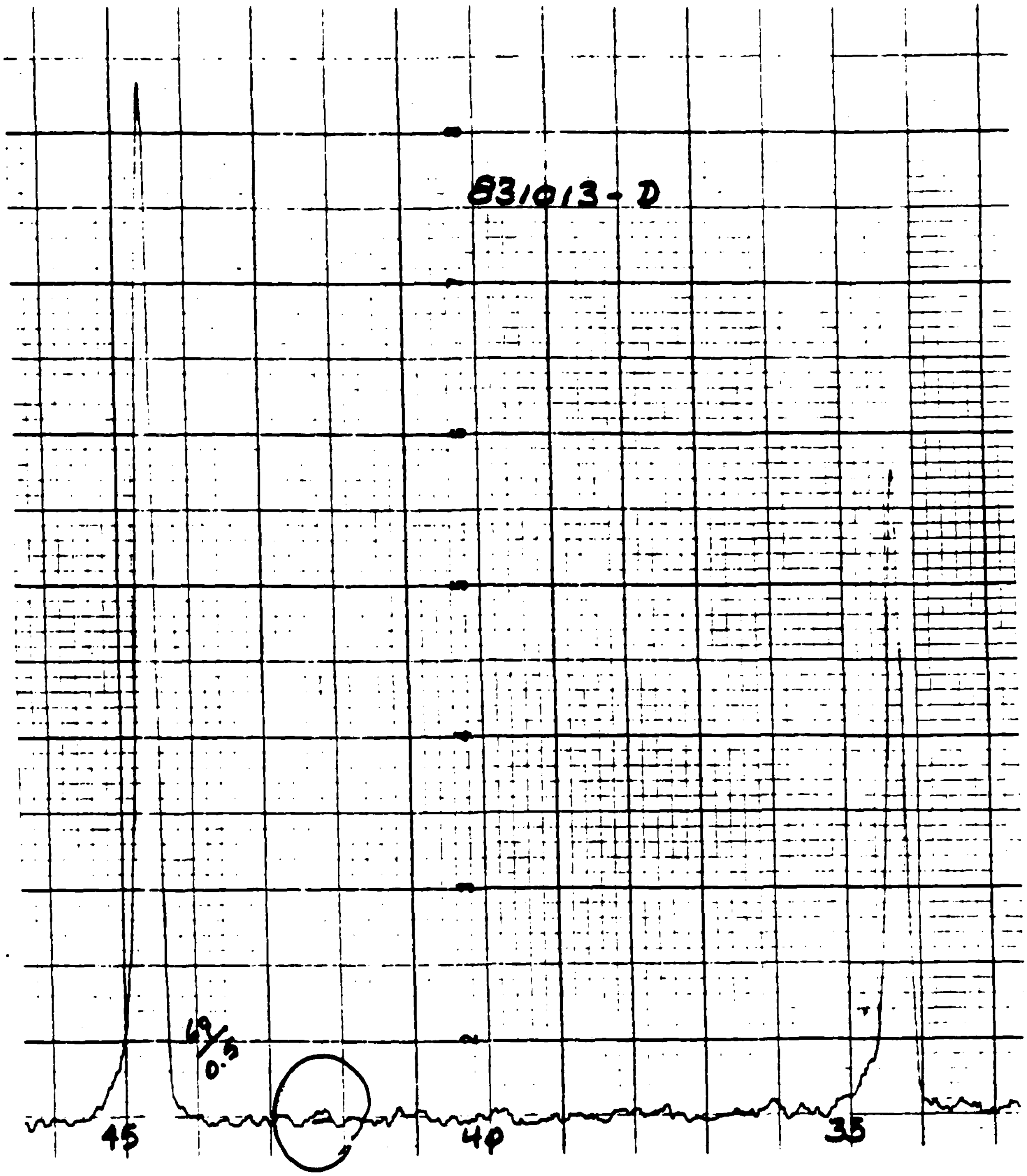
SURFACE AREA:

0.42 m^2/g

PARTICLE SIZE (MICROTRAC):

MV	17 μm
PH	29 μm
PM	15 μm
PS	6.5 μm

( ) See list of footnotes at end of Appendix A.



X-Ray Powder Diffraction Pattern for TiB_2 (Experiment XV)

[illegible] NO. XVI

ART REF. NO. 831013-E

MIX:

TiO_2 (1):	22.70 kg	60.0 %
B_4C (8):	8.43	22.3
C (3):	6.68	17.7

COMPOSITION:

	kg	moles	mole %
Ti:	13.75	287.1	14.4
B :	6.33	585.6	29.5
O :	8.92	557.5	28.0
C :	6.70	557.9	28.1

FIRING:

Temp Range: 2000 to 2020 °C

Weight of Feed: 18.6 kg.

Feed Rate: 5.9 kg/hr.

Weight of Product: 8.6 kg.

ANALYSIS:

Analytical Number: 1201

Ti (BTL, Colorimetric)	67.2	%
(SUNY, XRF)	71.1	%
B	24.62	%
C	7.79	%
O	0.55	%
N	0.067	%

Spectrographic

	n.a.	%
		%
		%
		%

X-RAY DIFFRACTION:

$$\frac{I\ (2\theta \cong 44.5°)}{I\ (2\theta \cong 42°)}$$ 86

0

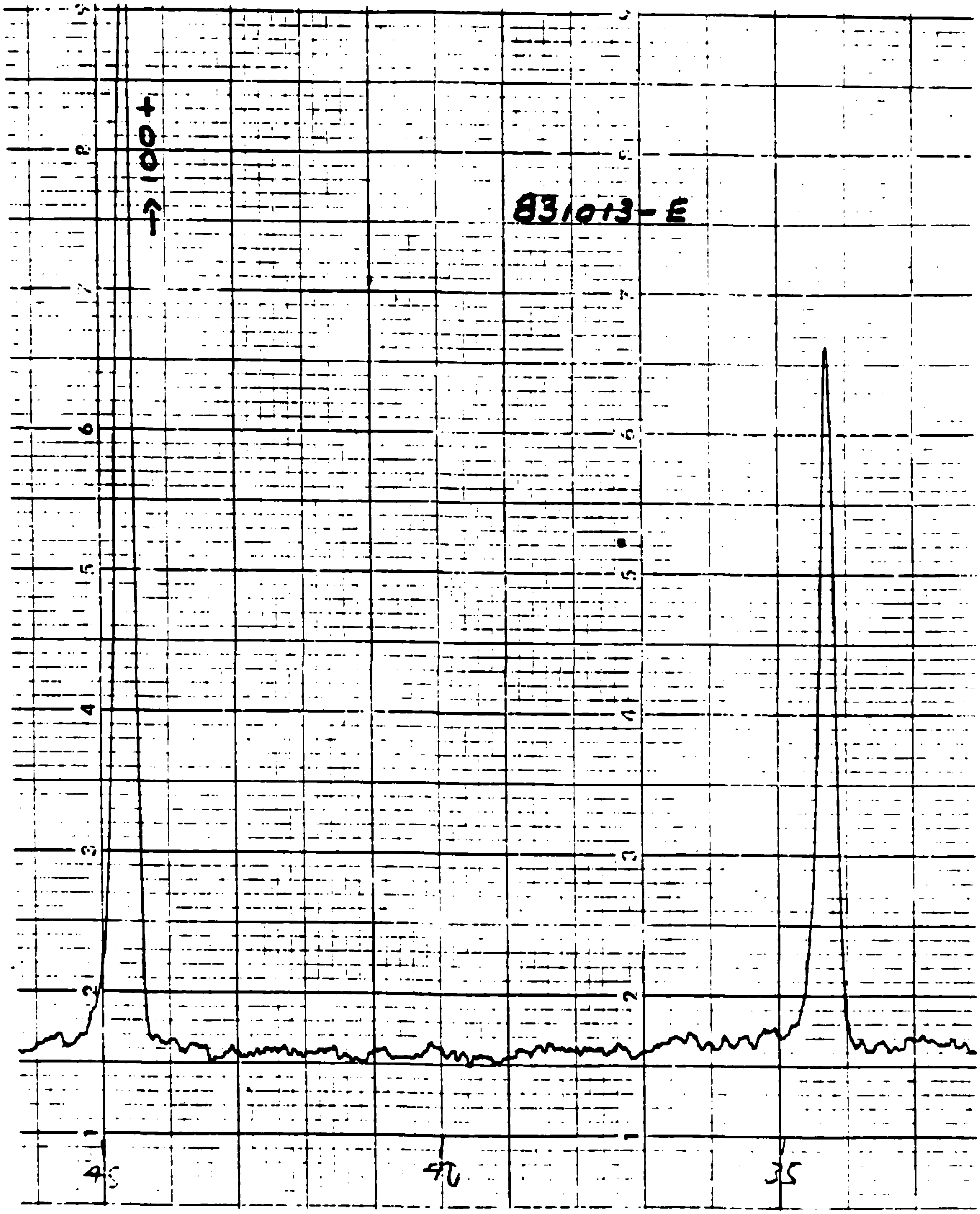
SURFACE AREA:

1.40 m^2/g

PARTICLE SIZE (MICROTRAC):

MV	16	μm
PH	30	μm
PM	10	μm
PS	4.1	μm

( ) See list of footnotes at end of Appendix A.



X-Ray Powder Diffraction Pattern for TiB_2 (Experiment XVI)

MIX:

TiO_2 (1):	22.70	kg	60.0	%
B_4C (8):	8.43		22.3	
C (3):	6.68		17.7	

COMPOSITION:

Ti:	13.75	kg	287.1	moles	14.4	mole %
B :	6.33		585.6		29.5	
O :	8.92		557.5		28.0	
C :	6.70		557.9		28.1	

FIRING:

Temp Range: 2100 to 2115 °C

Weight of Feed: 19.1 kg.

Feed Rate: 5.9 kg/hr.

Weight of Product: 11.8 kg.

ANALYSIS:

Analytical Number: 1201

Ti (BTL, Colorimetric)	65.08	%
(SUNY, XRF)	72.0	%
B	24.76	%
C	8.25	%
O	0.47	%
N	0.058	%
Spectrographic		
	n.a.	%
		%
		%
		%

X-RAY DIFFRACTION:

$$\frac{I\ (2\theta \cong 44.5°)}{I\ (2\theta \cong 42°)} \quad \frac{80}{0}$$

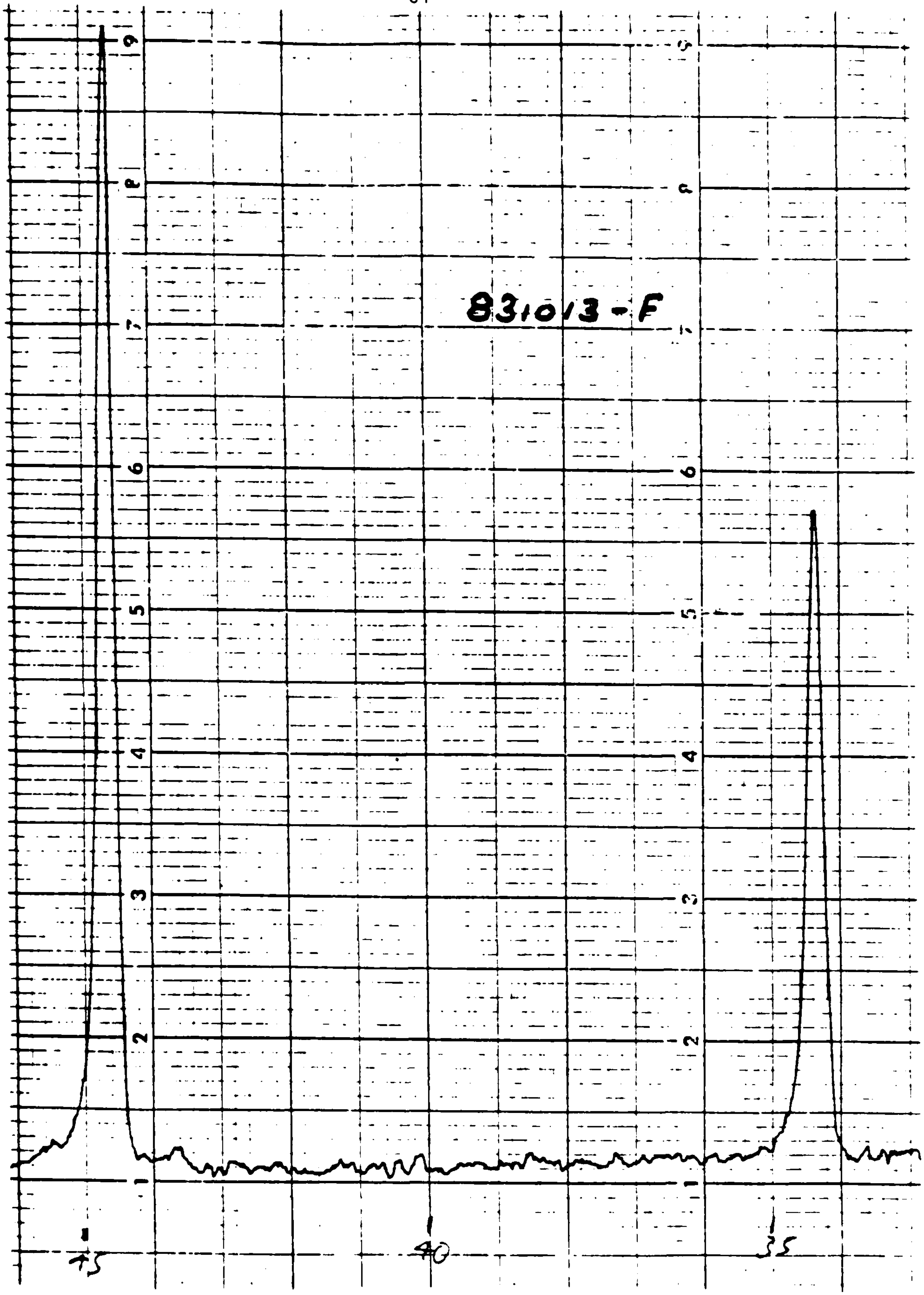
SURFACE AREA:

1.06 m^2/g

PARTICLE SIZE (MICROTRAC):

MV	14	μm
PH	25	μm
PM	11	μm
PS	5.2	μm

( ) See list of footnotes at end of Appendix A.



X-Ray Powder Diffraction Pattern for TiB_2 (Experiment XVII)

EXPT. NO. XVIII

ART REF. NO. 831108-A

MIX:

TiO_2 (1):	29.07 kg	64.6 %
B_4C (8):	11.70	26.0
C (3):	4.23	9.4

COMPOSITION:

Ti:	17.61 kg	367.6 moles	15.0 mole %
B :	8.79	813.1	33.3
O :	11.42	713.8	29.2
C :	6.58	548.3	22.4

FIRING:

Temp Range: 2000 to 2045 °C

Weight of Feed: 18.2 kg.

Feed Rate: n.a. kg/hr.

Weight of Product: 10.9 kg.

ANALYSIS:

Analytical Number: 1202

Ti (BTL, Colorimetric)	67.47	%
(SUNY, XRF)	69.6	%
B	22.02	%
C	0.62	%
O	2.02	%
N	1.80	%
Spectrographic		
Fe, Si	0.06	%
Ca	0.01	%
Others	< 0.006	%
		%

X-RAY DIFFRACTION:

$$\frac{I\ (2\theta \cong 44.5°)}{I\ (2\theta \cong 42°)} = \frac{70}{3}$$

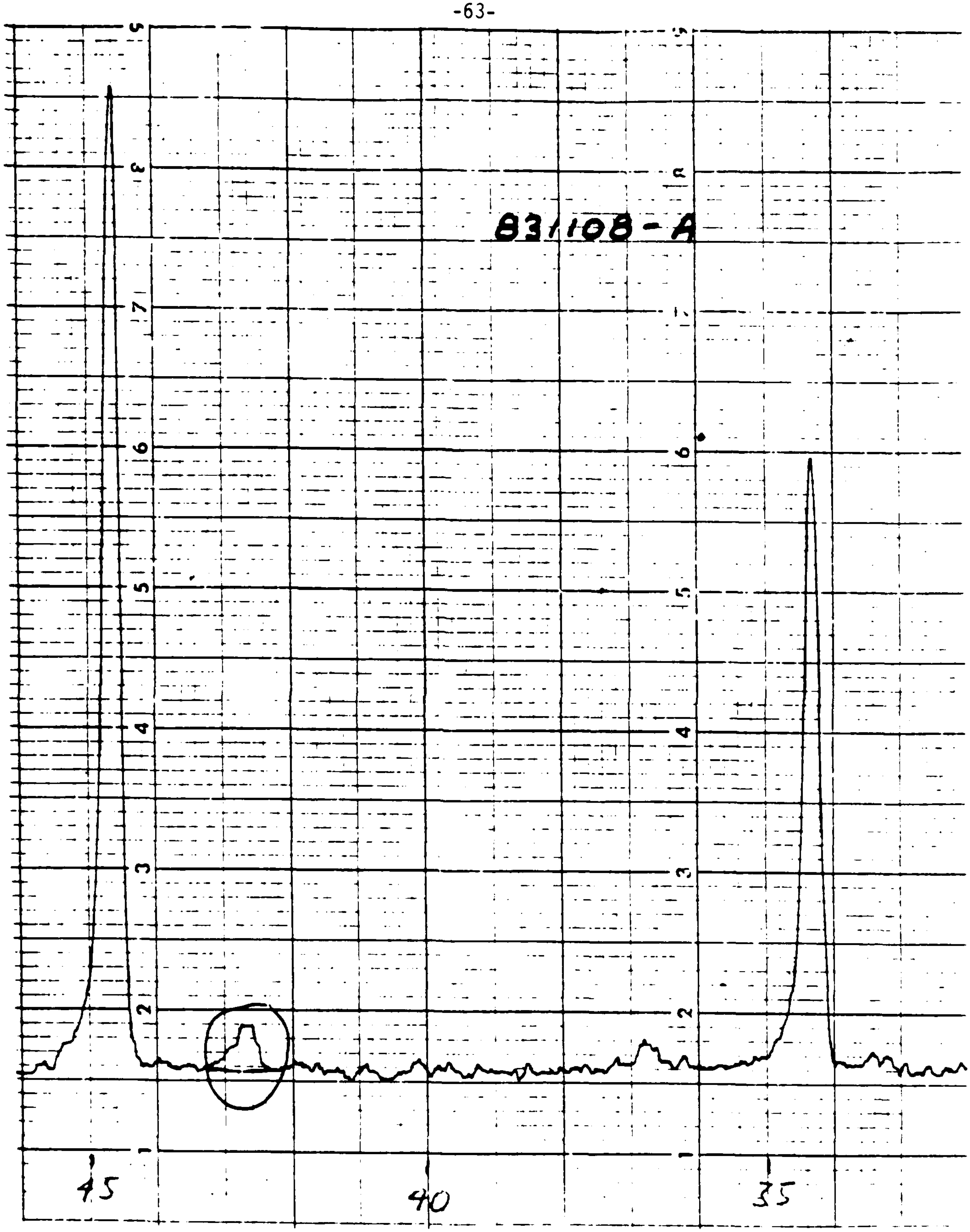
SURFACE AREA:

0.14 m^2/g

PARTICLE SIZE (MICROTRAC):

MV	22	µm
PH	43	µm
PM	16	µm
PS	5.9	µm

( ) See list of footnotes at end of Appendix A.



X-Ray Powder Diffraction Pattern for TiB_2 (Experiment XVIII)

ART REF. NO. 831108-B

MIX:

TiO_2 (1):	29.07 kg	64.6 %
B_4C (8):	11.70	26.0
C (3):	4.23	9.4

COMPOSITION:

	kg	moles	mole %
Ti:	17.61	367.6	15.0
B :	8.79	813.1	33.3
O :	11.42	713.8	29.2
C :	6.58	548.3	22.4

FIRING:

Temp Range: 2060 to 2125 °C

Weight of Feed: 18.2 kg.

Feed Rate: 4.1 kg/hr.

Weight of Product: 5.9 kg.

ANALYSIS:

Analytical Number: 1202

Ti (BTL, Colorimetric)	68.14	%
(SUNY, XRF)	70.6	%
B	24.36	%
C	1.07	%
O	1.35	%
N	0.62	%
Spectrographic		
Si	0.08	%
Fe	0.05	%
Zr	0.01	%
Others	<0.008	%

X-RAY DIFFRACTION:

$\frac{I\ (2\theta \cong 44.5^\circ)}{I\ (2\theta \cong 42^\circ)}$ 75

0

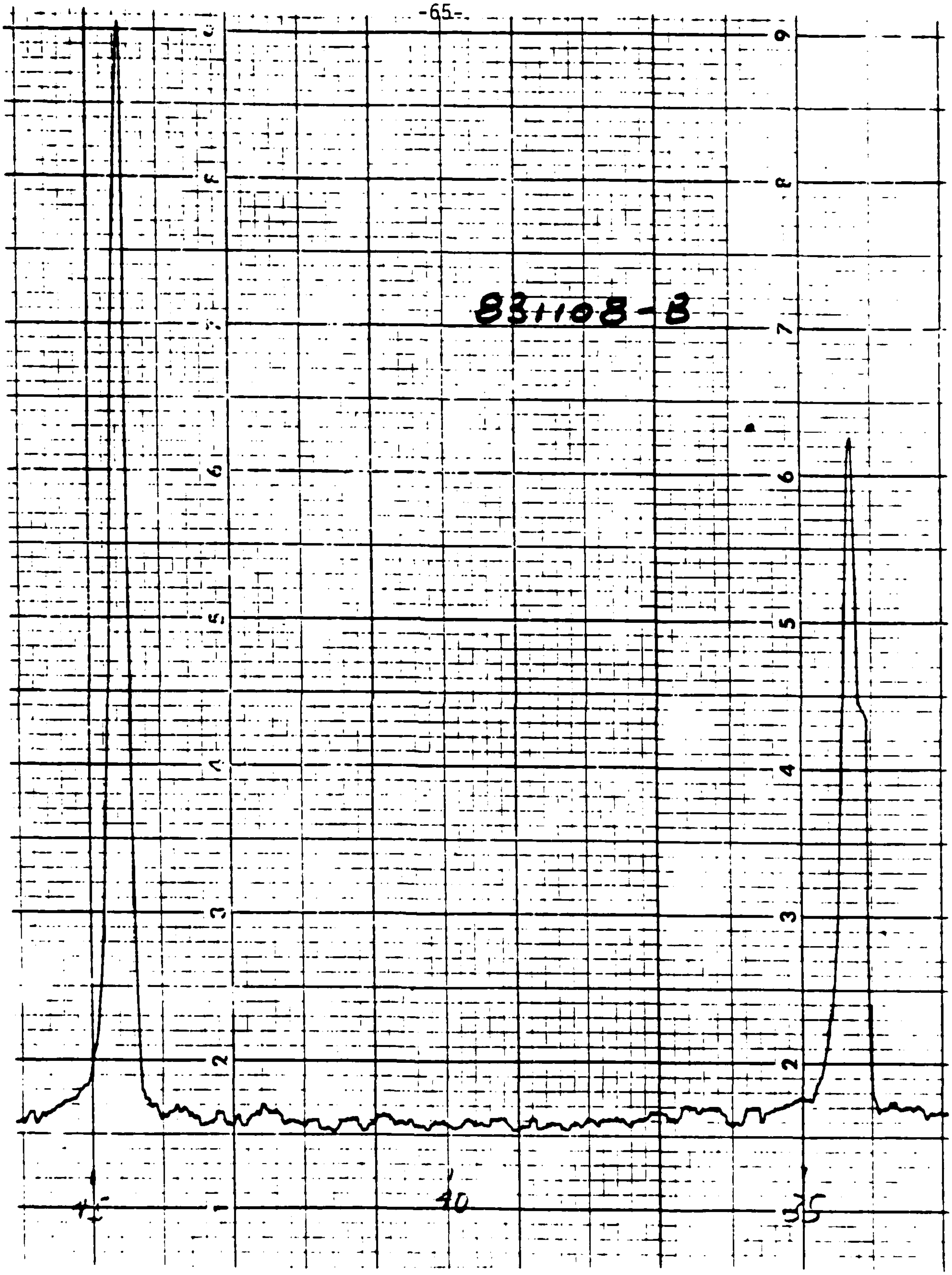
SURFACE AREA:

0.44 m^2/g

PARTICLE SIZE (MICROTRAC):

MV	18	μm
PH	30	μm
PM	13	μm
PS	5.1	μm

( ) See list of footnotes at end of Appendix A.



X-Ray Powder Diffraction Pattern for TiB_2 (Experiment XIX)

ART REF. NO. 831108-C

MIX:

TiO_2 (1):	28.76 kg	63.9 %
B_4C (8):	12.15	27.0
C (3):	6.35	14.1

COMPOSITION:

Ti:	17.43 kg	363.8 moles	13.7 mole %
B :	9.12	844.1	31.9
O :	11.30	706.4	26.7
C :	8.79	732.0	27.7

FIRING:

Temp Range: 2000 to 2025 °C

Weight of Feed: 18.2 kg.

Feed Rate: 4.5 kg/hr.

Weight of Product: 10.0 kg.

ANALYSIS:

Analytical Number: 1202

Ti (BTL, Colorimetric)	67.82	%
(SUNY, XRF)	70.3	%
B	24.45	%
C	0.57	%
O	1.21	%
N	0.13	%
Spectrographic		
Fe	0.1	%
Si	0.06	%
Others	< 0.006	%
		%

X-RAY DIFFRACTION:

$\frac{I\ (2\theta \cong 44.5°)}{I\ (2\theta \cong 42°)}$ 78.5 / 0

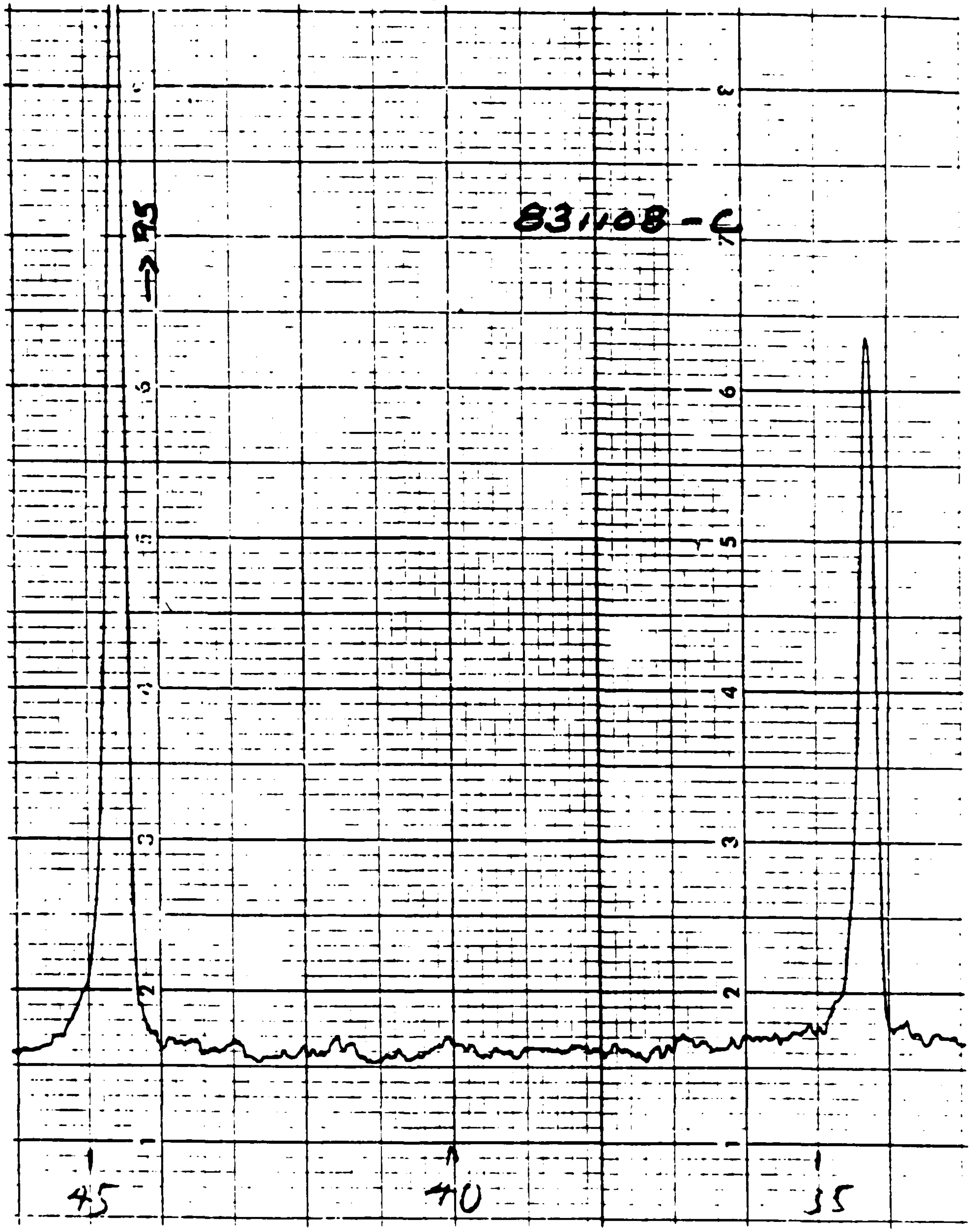
SURFACE AREA:

0.54 m^2/g

PARTICLE SIZE (MICROTRAC):

MV	19	µm
PH	35	µm
PM	14°	µm
PS	5.4	µm

( ) See list of footnotes at end of Appendix A.



X-Ray Powder Diffraction Pattern for TiB_2 (Experiment XX)

MIX:

TiO_2 (1):	28.76 kg	63.9 %
B_4C (8):	12.15	27.0
C (3):	6.35	14.1

COMPOSITION:

	kg	moles	mole
Ti:	17.43	363.8	13.7
B :	9.12	844.1	31.9
O :	11.30	706.4	26.7
C :	8.79	732.0	27.7

FIRING:

Temp Range: 2095 to 2120 °C

Weight of Feed: 18.6 kg.

Feed Rate: 2.3 kg/hr.

Weight of Product: 10.0 kg.

ANALYSIS:

Analytical Number: 1202

Ti (BTL, Colorimetric)	67.73	%
(SUNY, XRF)	71.3	%
B	24.28	%
C	0.31	%
O	1.07	%
N	0.48	%
Spectrographic		
Fe	0.06	%
Si	0.04	%
Others	< 0.004	%
		%

X-RAY DIFFRACTION:

$$\frac{I\ (2\theta \cong 44.5°)}{I\ (2\theta \cong 42°)}$$ 88 / 0

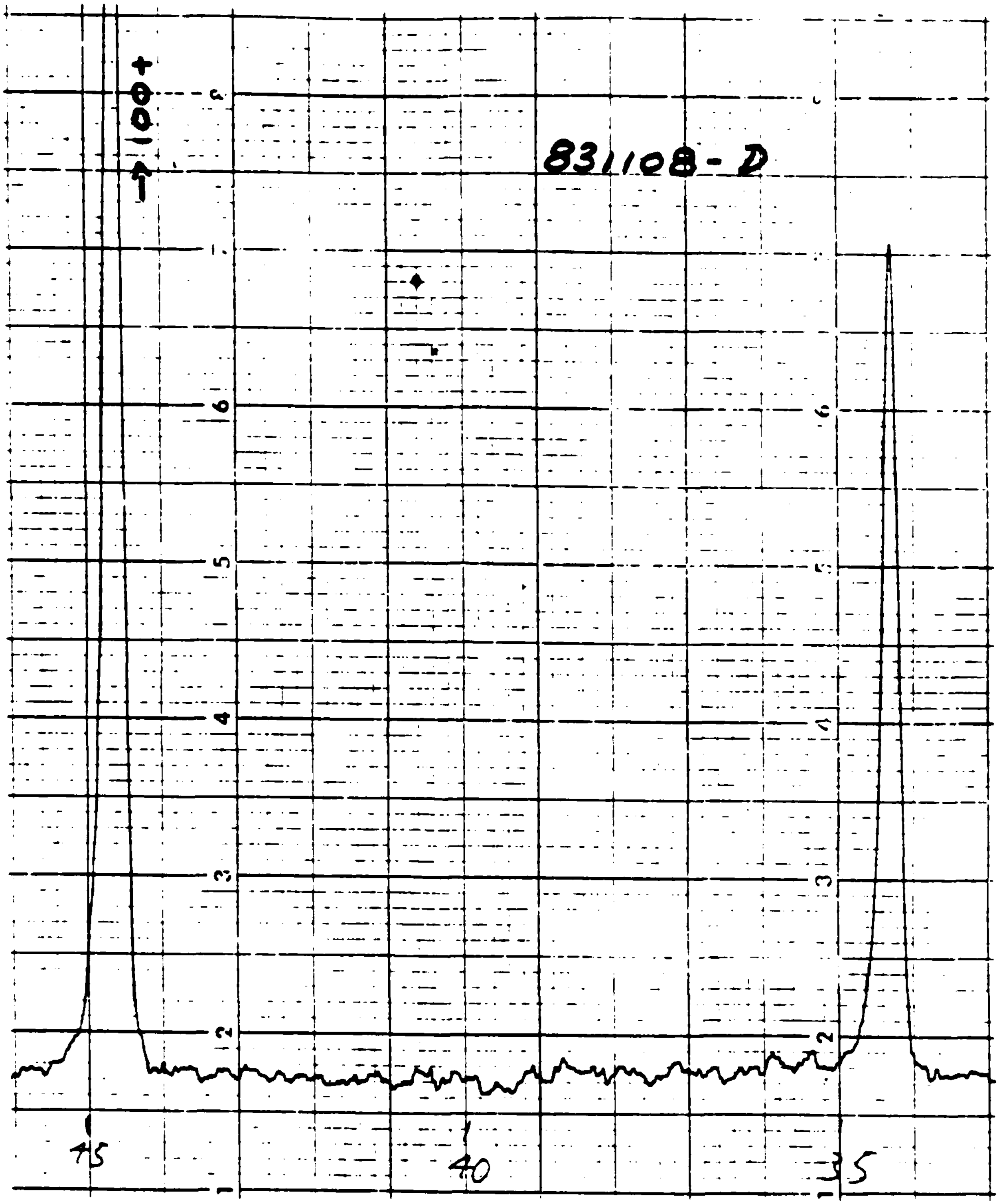
SURFACE AREA:

0.32 m^2/g

PARTICLE SIZE (MICROTRAC):

MV	17	µm
PH	29	µm
PM	14	µm
PS	5.8	µm

( ) See list of footnotes at end of Appendix A.



X-Ray Powder Diffraction Pattern for TiB_2 (Experiment XXI)

ART REF. NO. 831108-E

MIX:

TiO_2 (1):	28.44	kg	63.2	%
B_4C (8):	12.69		28.2	
C (3):	3.87		8.6	

COMPOSITION:

Ti:	17.23	kg	359.7	moles	14.5	mole %
B :	9.53		881.6		35.6	
O :	11.18		698.6		28.2	
C :	6.42		534.5		21.6	

FIRING:

Temp Range: 2000 to 2040 °C

Weight of Feed: 18.6 kg.

Feed Rate: 6.2 kg/hr.

Weight of Product: 7.3 kg.

ANALYSIS:

Analytical Number: 1202

Ti (BTL, Colorimetric)	65.37	%
(SUNY, XRF)	72.22	%
B	24.25	%
C	0.58	%
O	1.34	%
N	0.61	%
Spectrographic		
Fe	0.05	%
Si	0.02	%
Others	< 0.008	%
		%

X-RAY DIFFRACTION:

$$\frac{I\ (2\theta \cong 44.5^\circ)}{I\ (2\theta \cong 42^\circ)} \quad \frac{47}{0}$$

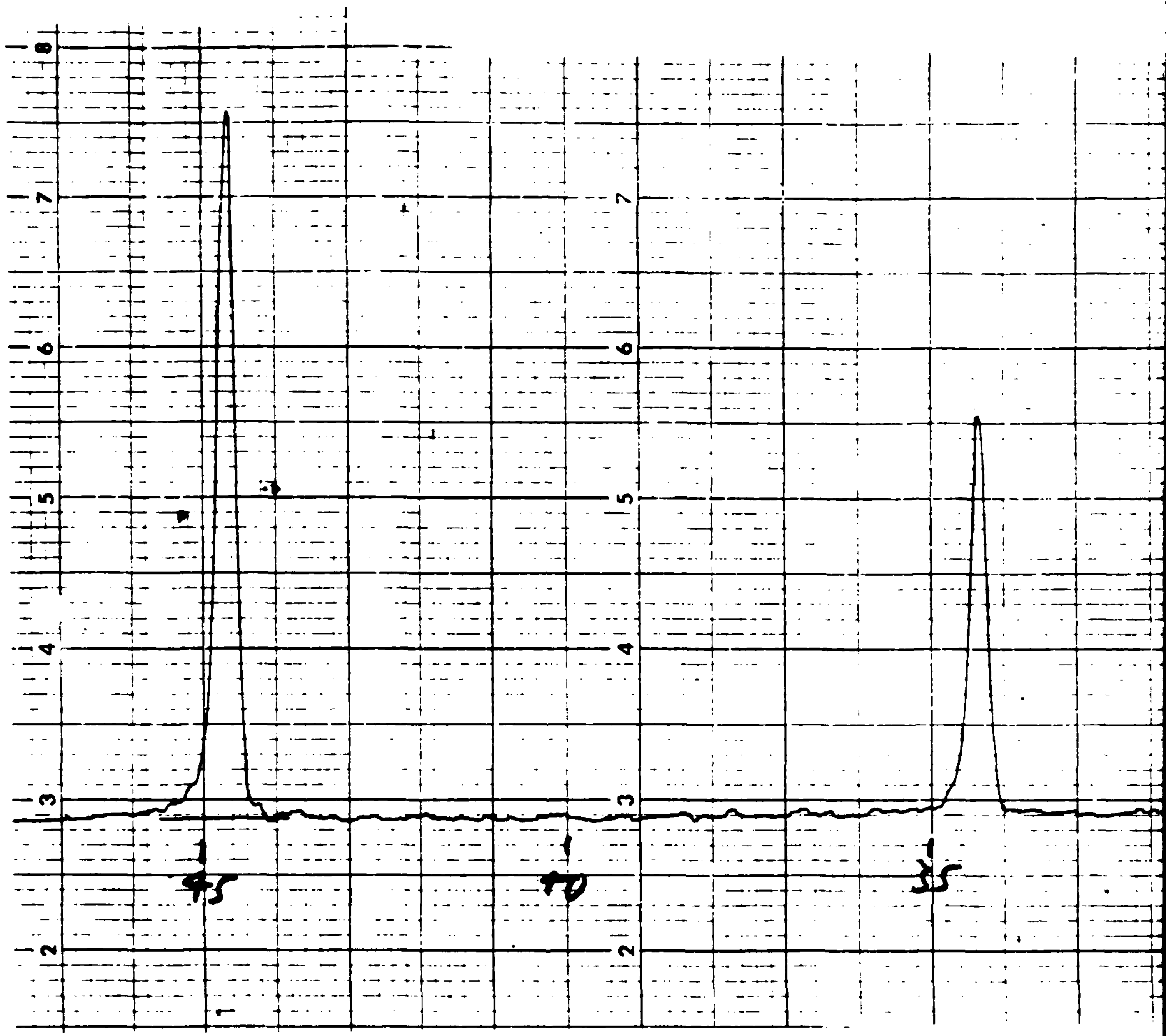
SURFACE AREA:

0.15 m^2/g

PARTICLE SIZE (MICROTRAC):

MV	22	μm
PH	52	μm
PM	15	μm
PS	5.9	μm

( ) See list of footnotes at end of Appendix A.



X-ray Powder Diffraction Pattern for TiB_2 (Experiment XXII)

MIX:

TiO_2 (1):	26.92 kg	61.7 %
B_4C (9):	11.35	26.0
C (3):	5.36	12.3

COMPOSITION:

Ti:	16.31 kg	340.6 moles	14.1 mole %
B :	8.15	753.8	31.1
O :	10.61	662.9	27.4
C :	7.97	663.9	27.4

FIRING:

Temp Range: 2050 to 2100 °C

Weight of Feed: 39.0 kg.

Feed Rate: 19.5 kg/hr.

Weight of Product: 15.0 kg.

ANALYSIS:

Analytical Number: 1293

Ti (BTL, Colorimetric)	66.57	%
(SUNY, XRF)	n.a.	%
B	29.09	%
C	3.37	%
O	0.38	%
N	0.055	%
Spectrographic		
Si	0.15	%
Fe	0.10	%
Others	< 0.02	%
		%

X-RAY DIFFRACTION:

$\frac{I\ (2\theta \cong 44.5°)}{I\ (2\theta \cong 42°)}$ n.a.

SURFACE AREA:

0.19, 0.74 (10) m^2/g

PARTICLE SIZE (MICROTRAC):

MV	16	14 (10)	μm
PH	29	26	μm
PM	14	12	μm
PS	5.6	4.9	μm

( ) See list of footnotes at end of Appendix A.

EXPT. NO. XXIV

ART REF. NO. 840123-F

MIX:

TiO_2 (1):	13.62 kg	62.8 %
B_4C (11):	5.45	25.1
C (3):	2.63	12.1

COMPOSITION:

	kg	moles	mole %
Ti:	8.17	170.6	13.6
B :	3.91	362.0	28.9
O :	5.45	340.6	27.2
C :	4.17	377.5	30.2

FIRING:

Temp Range: 2050 to 2100 °C

Weight of Feed: 21.7 kg.

Feed Rate: 9.1 kg/hr.

Weight of Product: 10.4 kg.

ANALYSIS:

Analytical Number: C-122

Ti (BTL, Colorimetric)	71.96	%
(SUNY, XRF)	71.63	%
B	30.02	%
C	0.26	%
O	1.44	%
N	0.05	%
Spectrographic		
Ca	0.3	%
		%
		%
Others	< 0.05	%

X-RAY DIFFRACTION:

$\frac{I\ (2\theta \cong 44.5°)}{I\ (2\theta \cong 42°)}$ 44 / 0

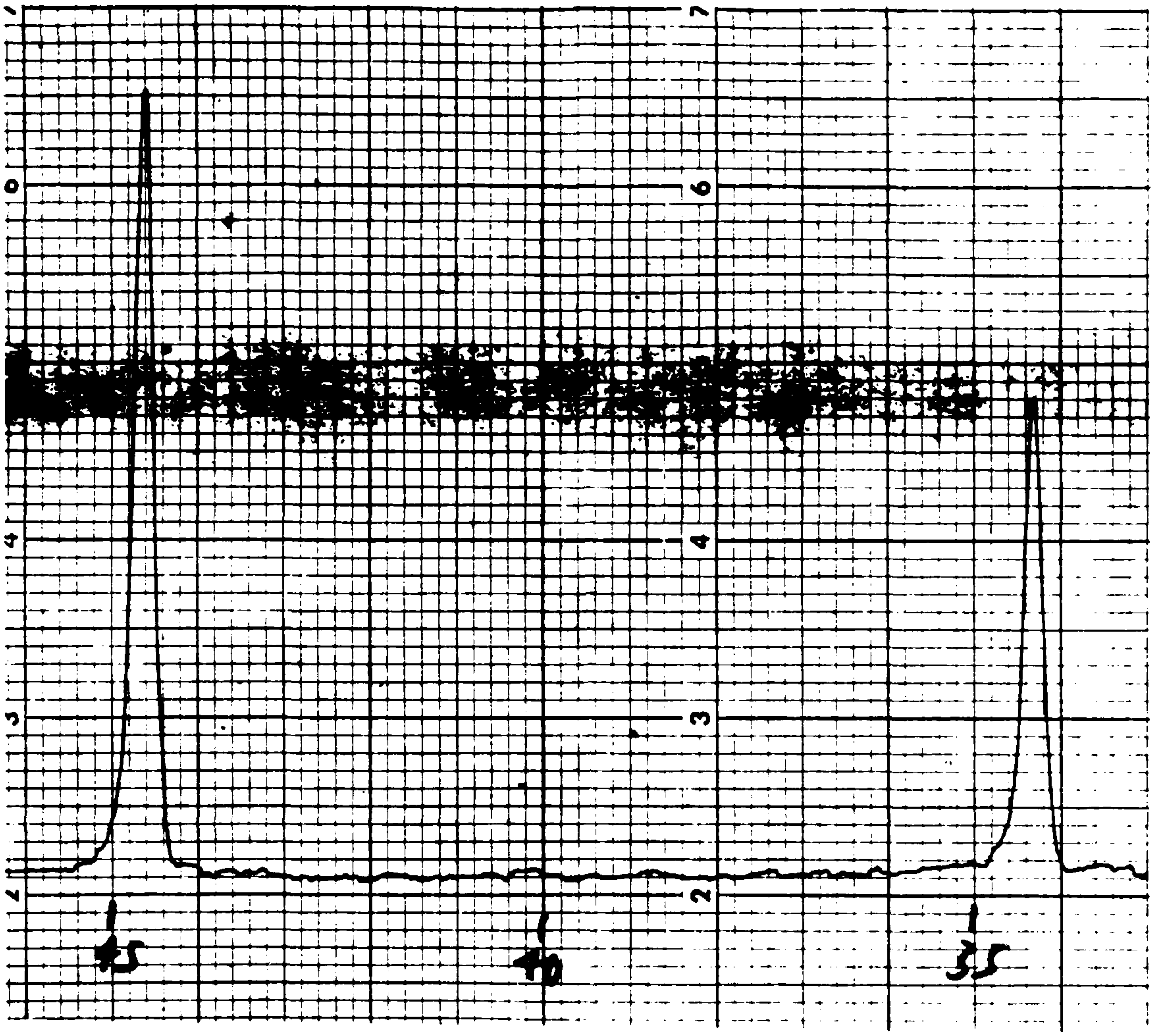
SURFACE AREA:

0.59 m^2/g

PARTICLE SIZE (MICROTRAC):

MV	16	µm
PH	32	µm
PM	13	µm
PS	4	µm

( ) See list of footnotes at end of Appendix A.



X-ray powder diffraction pattern for TiB_2 (Experiment XXIV)

EXPT. NO. XXV
ART REF. NO. 840124-F

MIX:

TiO_2 (1):	13.6 kg	61.5 %
B_4C ( ):	6.0	27.1
C (3):	2.5	11.3

COMPOSITION:

	kg	moles	mole %
Ti:	8.17	170.5	13.5
B :	4.30	398.5	31.7
O :	5.43	339.4	27.0
C :	4.20	350.0	27.8

FIRING:

Temp Range: 2050 to 2100 °C

Weight of Feed: 22.1 kg.

Feed Rate: 9.1 kg/hr.

Weight of Product: n.a. kg.

ANALYSIS:

Analytical Number: C-119, C-124

Ti (BTL, Colorimetric)	67.15	%
(SUNY, XRF)	69.49	%
B	29.71	%
C	0.24	%
O	3.45	%
N	0.051	%
Spectrographic		
Ca	0.4	%
Si	0.2	%
Fe	0.1	%
Others	< 0.05	%

X-RAY DIFFRACTION:

$\frac{I\ (2\theta \cong 44.5°)}{I\ (2\theta \cong 42°)}$ 39 / 0

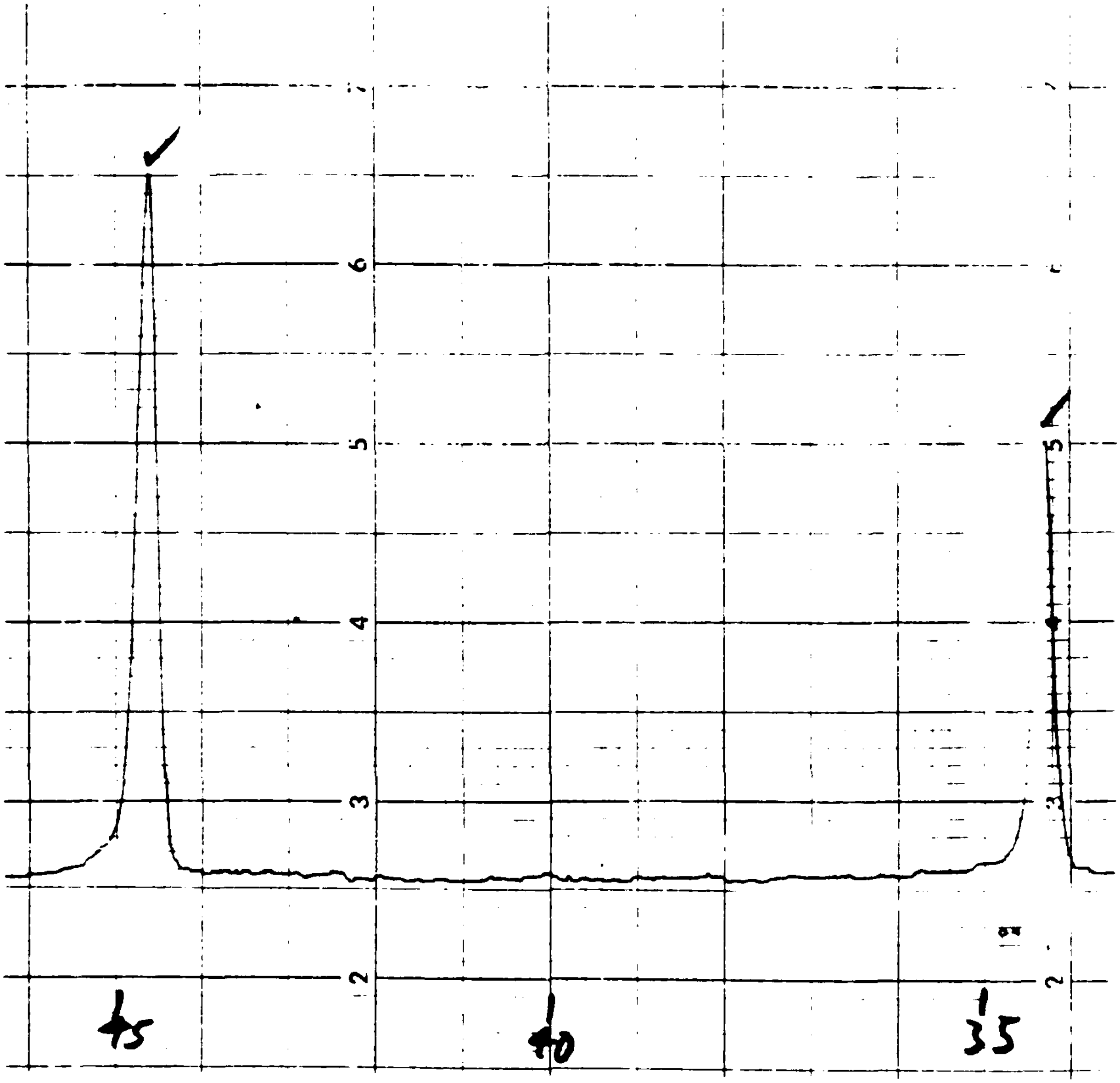
SURFACE AREA:

0.50 m^2/g

PARTICLE SIZE (MICROTRAC):

MV	27	μm
PH	49	μm
PM	22	μm
PS	8	μm

( ) See list of footnotes at end of Appendix A.



X-ray Powder Diffraction Pattern for TiB_2 (Experiment XXV)

EXPT. NO. XXVI

ART REF. NO. 840413-AFR

MIX:

TiB_2 (12):	88.53 kg	91.7 %
B_4C (8):	4.36	4.5
H_3BO_3 (13):	3.27	3.4
C (14):	0.36	0.4

COMPOSITION:

	kg	moles	mole %
Ti:	60.1	1254.9	34.1
B:	23.0	2127.7	57.9
O:	2.59	161.9	4.4
C:	1.59	132.4	3.6
N:	0.2	Neglected	Neglected

FIRING:

Temp Range: 2050 to 2075 °C

Weight of Feed: 37.7 kg.

Feed Rate: 25 kg/hr.

Weight of Product: 26.8 kg.

ANALYSIS:

Analytical Number:	C-359 (16)		
Ti (BTL, Colorimetric)	65.56	58.47	%
(SUNY, XRF)	n.a.	70.75	%
B	33.27	34.90	%
C	1.17	2.39	%
O	0.50	2.00	%
N	0.44	0.28	%
Spectrographic			
Ca	0.3	0.2	%
Fe	0.1	0.1	%
			%
Others	< 0.05		%

X-RAY DIFFRACTION:

$\frac{I\ (2\theta \cong 44.5°)}{I\ (2\theta \cong 42°)}$ 42 / 0

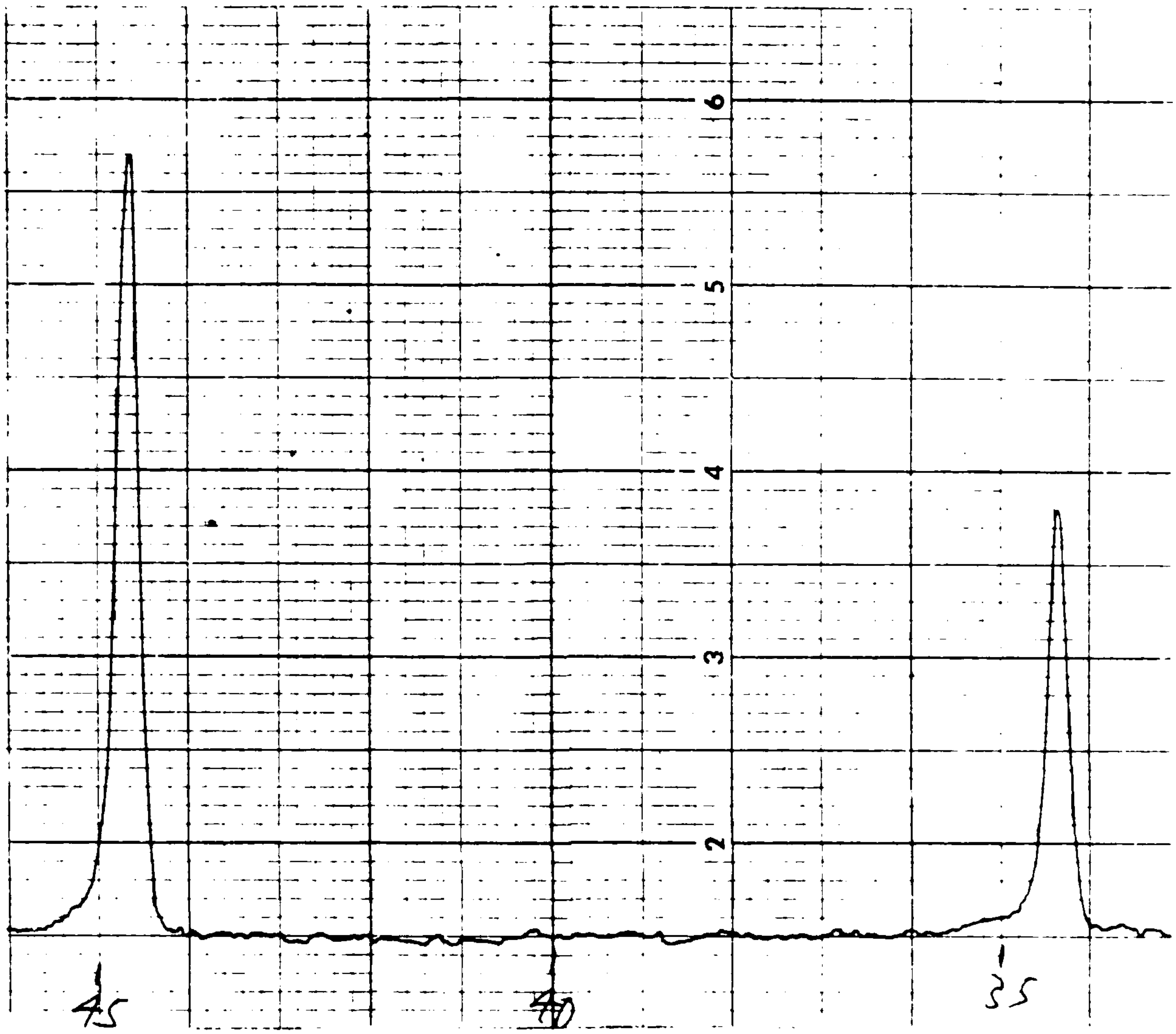
SURFACE AREA:

0.49 m^2/g

PARTICLE SIZE (MICROTRAC):

MV	21	μm
PH	41	μm
PM	15	μm
PS	5.5	μm

( ) See list of footnotes at end of Appendix A.



X-ray Powder Diffraction Pattern for TiB_2 (Experiment XXVI)

EXPT. NO. XXVII

ART REF. NO. 840416-AFR

MIX:

Same as XXVI, Unfired Portion fired at a later time.

COMPOSITION:

FIRING:

Temp Range:	2040	to 2080	°C
Weight of Feed:	55.0		kg.
Feed Rate:	18		kg/hr.
Weight of Product:	37.3		kg.

ANALYSIS:

Analytical Number:	C-359 (16)		
Ti (BTL, Colorimetric)	65.31	65.87	%
(SUNY, XRF)	n.a.	71.30	%
B	31.69	32.19	%
C	0.72	0.70	%
O	0.41	0.74	%
N	0.43	0.31	%
Spectrographic			
Ca	0.1	0.2	%
Fe	0.1	0.15	%
Si	0.006	0.2	%
Others	< 0.05		%

X-RAY DIFFRACTION:

$\frac{I\ (2\theta \cong 44.5°)}{I\ (2\theta \cong 42°)}$ 38 / 0

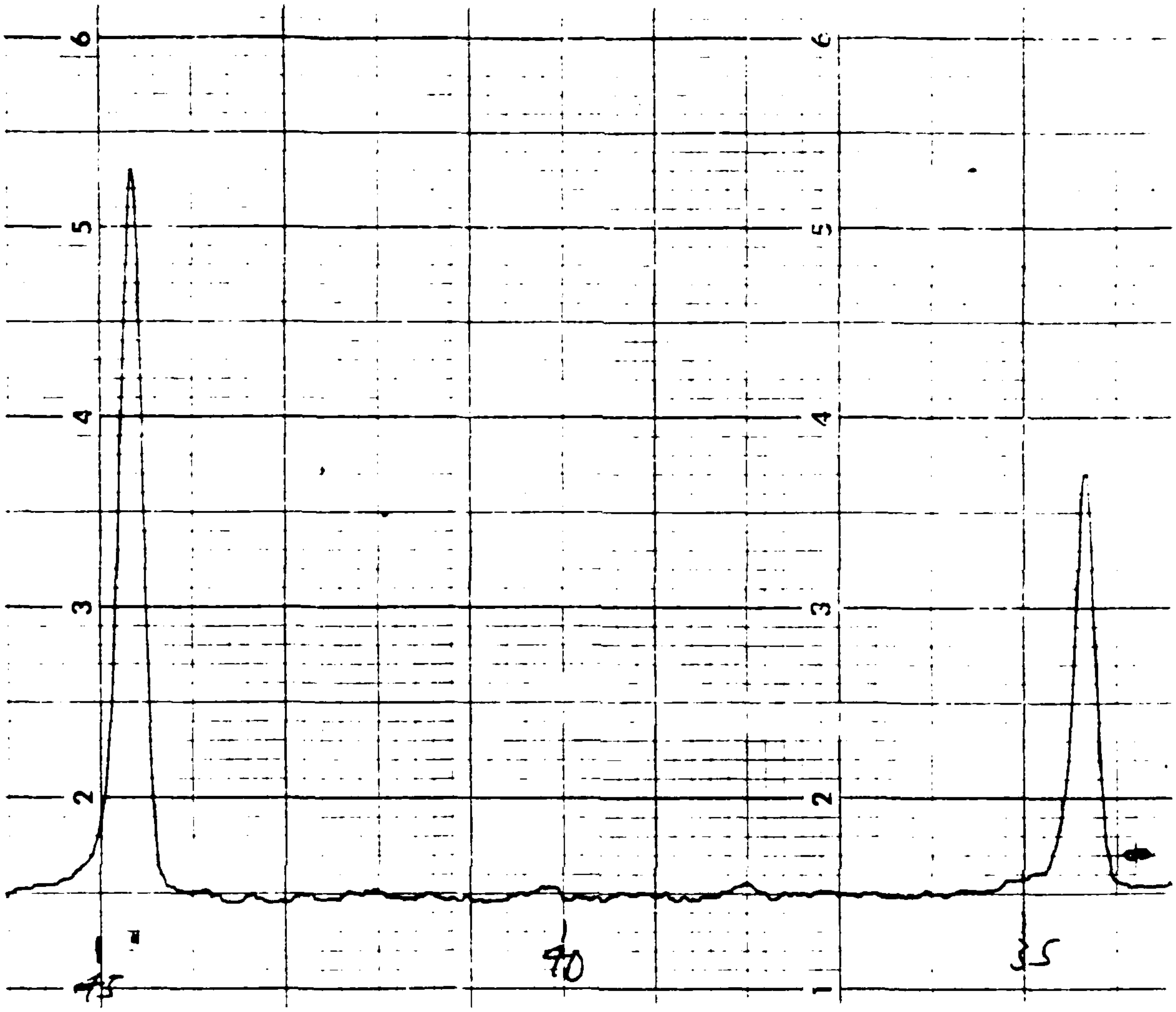
SURFACE AREA:

0.59 m^2/g

PARTICLE SIZE (MICROTRAC):

MV	n.a.	μm
PH	n.a.	μm
PM	n.a.	μm
PS	n.a.	μm

( ) See list of footnotes at end of Appendix A.



X-Ray Powder Diffraction Pattern of TiB_2 (Experiment XXVII)

EXPT. NO. XXVIII

ART REF. NO. 840412

MIX:

TiO_2 (1):	3.15 kg	63.4 %	
B_4C (8):	1.10	22.2	
C (3):	0.715	14.4	

COMPOSITION:

	kg	moles	mole %
Ti:	1.89	39.5	14.3
B:	0.83	76.8	27.8
O:	1.26	78.8	28.6
C:	0.97	80.8	29.3

FIRING:

Temp Range: 2025 to 2075 °C

Weight of Feed: 5.0 kg.

Feed Rate: 11.4 kg/hr.

Weight of Product: 2.5 kg.

ANALYSIS:

Analytical Number:	C-339	(16)	
Ti (BTL, Colorimetric)	64.60	46.57	%
(SUNY, XRF)	n.a.	63.55	%
B	22.53	35.12	%
C	4.53	11.88	%
O	2.06	1.90	%
N	0.19	0.15	%
Spectrographic			
Fe	0.2	>1.0	%
Ca	0.2	0.3	%
Si	0.1	0.2	%
Others	<0.05		%

X-RAY DIFFRACTION:

$$\frac{I\ (2\theta \cong 44.5°)}{I\ (2\theta \cong 42°)} \quad \frac{36}{6}$$

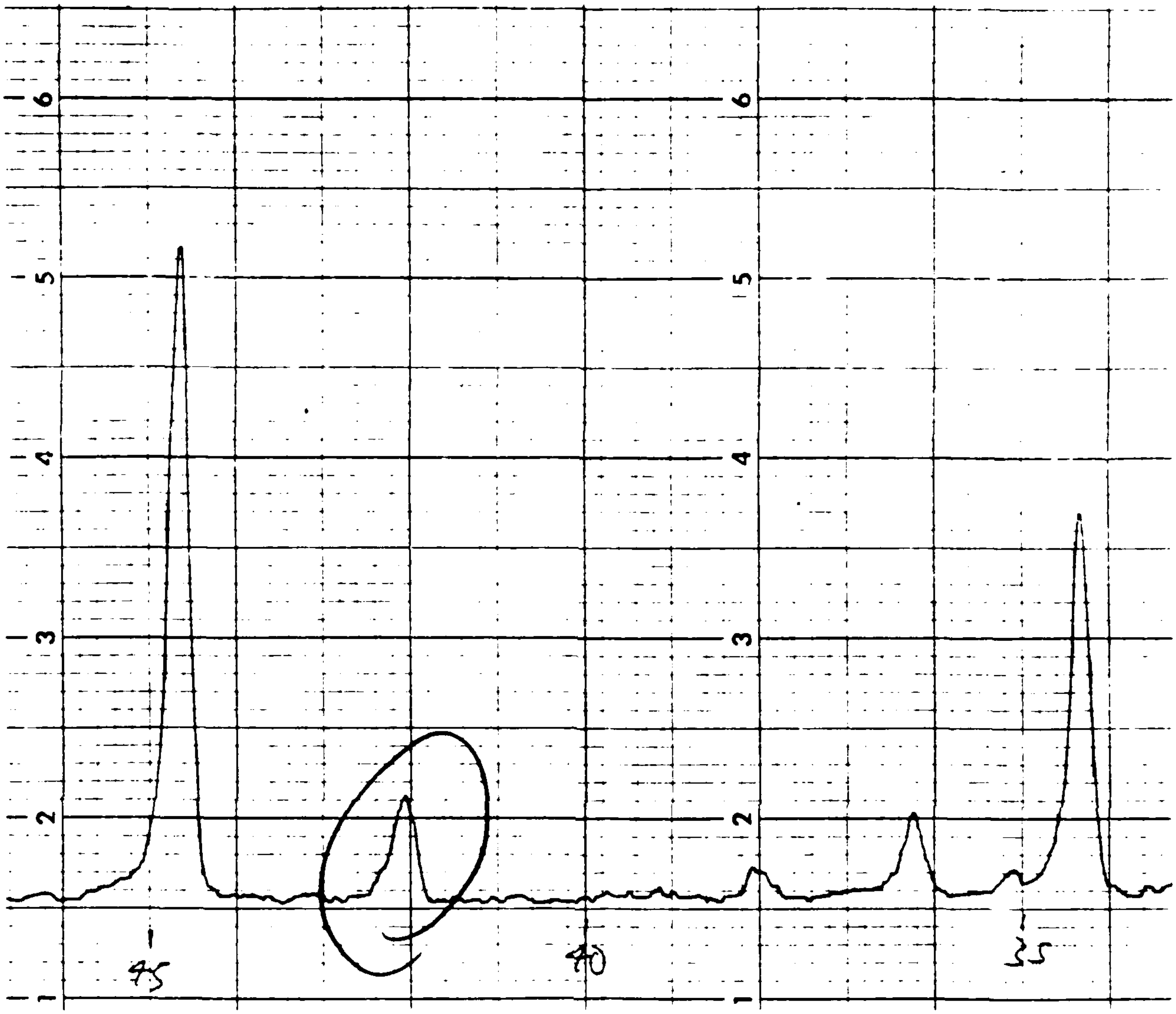
SURFACE AREA:

1.22 m^2/g

PARTICLE SIZE (MICROTRAC):

MV	25	μm
PH	53	μm
PM	19	μm
PS	4.6	μm

( ) See list of footnotes at end of Appendix A.



X-Ray Powder Diffraction Pattern of TiB_2 (Experiment XXVIII)

EXPT. NO. XXIX

ART REF. NO. 840501-P

MIX:

TiO_2 (1):	104.0 kg	62.4 %
B_4C (15):	41.8	25.1
C (3):	20.9	43.6

COMPOSITION:

	kg	moles	mole %
Ti:	62.40	1302.7	13.9
B :	29.69	2746.5	29.2
O :	41.60	2600.0	27.7
C :	33.01	2750.8	29.3

FIRING:

Temp Range: 2045 to 2100 °C

Weight of Feed: 129 kg.

Feed Rate: 13.25 kg/hr.

Weight of Product: 70.5 kg.

ANALYSIS:

Analytical Number:	C-437 (16)	C-559	
Ti(BTL,Colorimetric)	65.92	58.80	%
(SUNY, XRF)	n.a.	n.a.	%
B	29.96	31.50	%
C	0.37	2.67	%
O	3.01	5.91	%
N	0.03	0.07	%
Spectrographic			
Ca	0.2	0.2	%
Fe	>1.0	>1.0	%
Si	0.06	0.2	%
Others	< 0.02		%

X-RAY DIFFRACTION:

$\frac{I\ (2\theta \cong 44.5°)}{I\ (2\theta \cong 42°)}$ 47 / < 1

SURFACE AREA:

0.33 m^2/g

PARTICLE SIZE (MICROTRAC):

MV	n.a.	μm
PH	n.a.	μm
PM	n.a.	μm
PS	n.a.	μm

( ) See list of footnotes at end of Appendix A.

X-Ray Powder Diffraction Pattern of TiB_2 (Experiment XXIX)

ART REF. NO. 840412-R

MIX:

Product from Expt. XXVIII

COMPOSITION:

Ti :	55.59	weight % (18)	25.0 Mole%
B :	28.83		57.5
C :	8.20		14.7
O :	1.98		2.7
N :	0.17		neglected

FIRING:

Temp Range: 2050 to 2100 °C

Weight of Feed: n.a. kg.

Feed Rate: n.a. kg/hr.

Weight of Product: n.a. kg.

ANALYSIS:

Analytical Number: C-560

Ti(BTL,Colorimetric)	65.30	%
(SUNY, XRF)	n.a.	%
B	29.67	%
C	2.75	%
O	3.43	%
N	0.52	%
Spectrographic		
Fe	>1.0	%
Si	0.2	%
W	0.06	%
Others	≤0.1	%

X-RAY DIFFRACTION:

$\frac{I\ (2\theta \cong 44.5°)}{I\ (2\theta \cong 42°)}$ ______

SURFACE AREA:

0.50 m^2/g

PARTICLE SIZE (MICROTRAC):

MV	30	μm
PH	59	μm
PM	24	μm
PS	7.4	μm

( ) See list of footnotes at end of Appendix A.

FOOT NOTES TO APPENDIX A

(1) N. L. Industries, Titanox 3030

(2) Lot 830218

(3) R. T. Vanderbilt, Thermax, Lot C-420

(4) data not available

(5) H. C. Starck, Lot 1

(6) Portion of TiB_2 (830812) refired

(7) H. C. Starck, Lot 830921

(8) ART, Lot 831010

(9) ART, Lot 831209

(10) Sample hammer milled briefly to break up agglomerates.

(11) ART, Lot 831215

(12) ART, Lot 840328

(13) U. S. Borax

(14) Carbon Residue from sugar binder

(15) ART, Lot 840413

(16) Analysis done on +200 and -200 mesh fractions (left and right columns respectively).

(17) Analyses by Martin Marietta Laboratory showed 71.54% Ti, 26.78% B.

(18) Average of analyses of 2 fractions of XXVIII

APPENDIX B

CHARACTERIZATION OF REACTANTS

Material	Supplier	Lot/Designation
Boric Acid	U. S. Borax	Granular
Boron Carbide	A.R.T.	1217
	A.R.T.	830218
	A.R.T.	831010, VIII-46
	A.R.T.	831215
	A.R.T.	C-401-3
	Starck	Lot 1
	Starck	830921, VII-64
Carbon	R. T. Vanderbilt	N-990, VIII-128
	R. T. Vanderbilt	N-990, IX-37
Corn Syrup	Best Foods	No.-99530, VIII-114
Titanium Diboride	A.R.T.	840328, IX-146
Titanium Dioxide	N. L. Industries	Titanox 3030, VII-118
		Titanox 3030, X-37

ANALYSIS OF REACTANTS

Material	Boric Acid
Supplier	U. S. Borax
Product Designation	Granular
ART Lot No.	
ART Anal. No.	

( )

Quantities required for one experiment did not justify characterization. See Experiments XXVI and XXVII.

( ) Foot notes summarized at end of Appendix B

ANALYSIS OF REACTANTS

Material	Boron Carbide
Supplier	ART
Product Designation	830218
ART Lot No.	830218
ART Anal. No.	404 and 406

Total boron	77.77, 76.90	Average 77.34
Total carbon	20.22%	

Particle Size (Microtrac)	MV	8.0μm
	90%	15.6
	50%	6.3
	10%	2.6

( ) Foot notes summarized at end of Appendix B

ANALYSIS OF REACTANTS

Material	Boron Carbide
Supplier	ART
Product Designation	831010
ART Lot No.	831010
ART Anal. No.	1062

Total Boron	75.1
Total Carbon (1)	23.41
Oxygen (1)	0.23
Nitrogen (1)	0.01
Spectrographic (1)	
Iron	0.1
Others	≤ 0.06

( ) Foot notes summarized at end of Appendix B

ANALYSIS OF REACTANTS

Material	Boron Carbide
Supplier	ARI
Product Designation	831209
ART Lot No.	831209, VIII-96
ART Anal. No.	1217

Total Boron	71.8
Total Carbon	22.7
Oxygen (1)	0.8
Nitrogen (1)	0.02

( ) Foot notes summarized at end of Appendix B

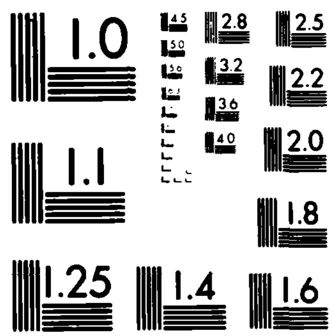
ANALYSIS OF REACTANTS

Material	Boron Carbide
Supplier	ART
Product Designation	831215
ART Lot No.	831215
ART Anal. No.	1299

Total Boron	75.56 %
Boric Oxide	0.71 %
Free Carbon	5.86 %

( ) Foot notes summarized at end of Appendix B

AD-A170 085 A PROCESS STUDY FOR MANUFACTURING ULTRA-FINE TITANIUMN 2/2
DIBORIDE POWDER(U) ADVANCED REFRACTORY TECHNOLOGIES INC
BUFFALO NY P T SHAFFER MAY 86 MTL-TR-86-18
UNCLASSIFIED DAAG46-83-C-0171 F/G 7/1 NL



MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS 1963 A

ANALYSIS OF REACTANTS

Material	Boron Carbide
Supplier	A.R.T.
Product Designation	840413
ART Lot No.	X-37
ART Anal. No.	C-401-3

Total Boron	71.03 %

( ) Foqt notes summarized at end of Appendix B

ANALYSIS OF REACTANTS

Material	Boron Carbide
Supplier	H. C. Starck
Product Designation	Lot 1
ART Lot No.	VII-79
ART Anal. No.	

Total Boron	72.0 %

( ) Foot notes summarized at end of Appendix B

ANALYSIS OF REACTANTS

Material	Boron Carbide
Supplier	H. C. Starck
Product Designation	
ART Lot No.	830921
ART Anal. No.	984

Total Boron	72.78, 73.01	Average 72.90
Soluble Boron		0.67
Total Carbon (1)		20.09
Free Carbon		6.53
Oxygen		2.02
Nitrogen (1)		0.29
Spectrographic (1)		
silicon		0.4
iron		0.2
calcium		0.1
others		≤ 0.04

( ) Foot notes summarized at end of Appendix B

ANALYSIS OF REACTANTS

Material	Carbon
Supplier	R. T. Vanderbilt
Product Designation	N-990
ART Lot No.	831230, VIII-128
ART Anal. No.	1332, C-100

Total Carbon (1)	99.0 %
Moisture (110°C)	0.022 %
Ash (1000°C, 3 hr.)	N.D.
Spectrographic (1)	all < 0.0006 %

( ) Foot notes summarized at end of Appendix B

ANALYSIS OF REACTANTS

Material	Carbon
Supplier	R. T. Vanderbilt
Product Designation	N-990
ART Lot No.	X-37
ART Anal. No.	C-420

Total Carbon	98.97 %
Spectrographic (1)	
Na	0.06
W	0.02
Others	< 0.002

( ) Foot notes summarized at end of Appendix B

ANALYSIS OF REACTANTS

Material	Corn Syrup
Supplier	Best Foods
Product Designation	No. 99530
ART Lot No.	VIII-114
ART Anal. No.	1327, 1333

Density	1.36 g/cm^3
Carbon Residue (6 hrs. to 450°C)	9.31 %

( ) Foot notes summarized at end of Appendix B

ANALYSIS OF REACTANTS

Material	Titanium Diboride
Supplier	ART (2)
Product Designation	840328
ART Lot No.	840328
ART Anal. No.	C-260

Titanium (1)	67.90 %
Boron	26.0
Carbon (1)	1.77
Oxygen (1)	2.93
Nitrogen (1)	0.25

( ) Foot notes summarized at end of Appendix B

ANALYSIS OF REACTANTS

Material	Titanium Dioxide
Supplier	N. L. Industries
Product Designation	Titanox 3030
ART Lot No.	830928
ART Anal. No.	985

Titanium	60.39 % (59.95% theoretical)
Calcium	0.2
Chromium	0.002
Zirconium	0.002
Others	≤ 0.0006

( ) **Foot notes summarized at end of Appendix B**

ANALYSIS OF REACTANTS

Material	Titanium Dioxide
Supplier	N. L. Industries
Product Designation	Titanox 3030
ART Lot No.	840413, X-37
ART Anal. No.	C-420

Ti	59.76 %
Spectrographic	
Ca	0.2
W	0.02
B	0.01
Others	< 0.01

( ) Foot notes summarized at end of Appendix B

FOOT NOTES FOR APPENDIX B

(1) Analysis by Buffalo Testing Laboratories

(2) Produced by combining various lots of TiB_2

APPENDIX C

CHARACTERIZATION OF COMMERCIAL TITANIUM DIBORIDE

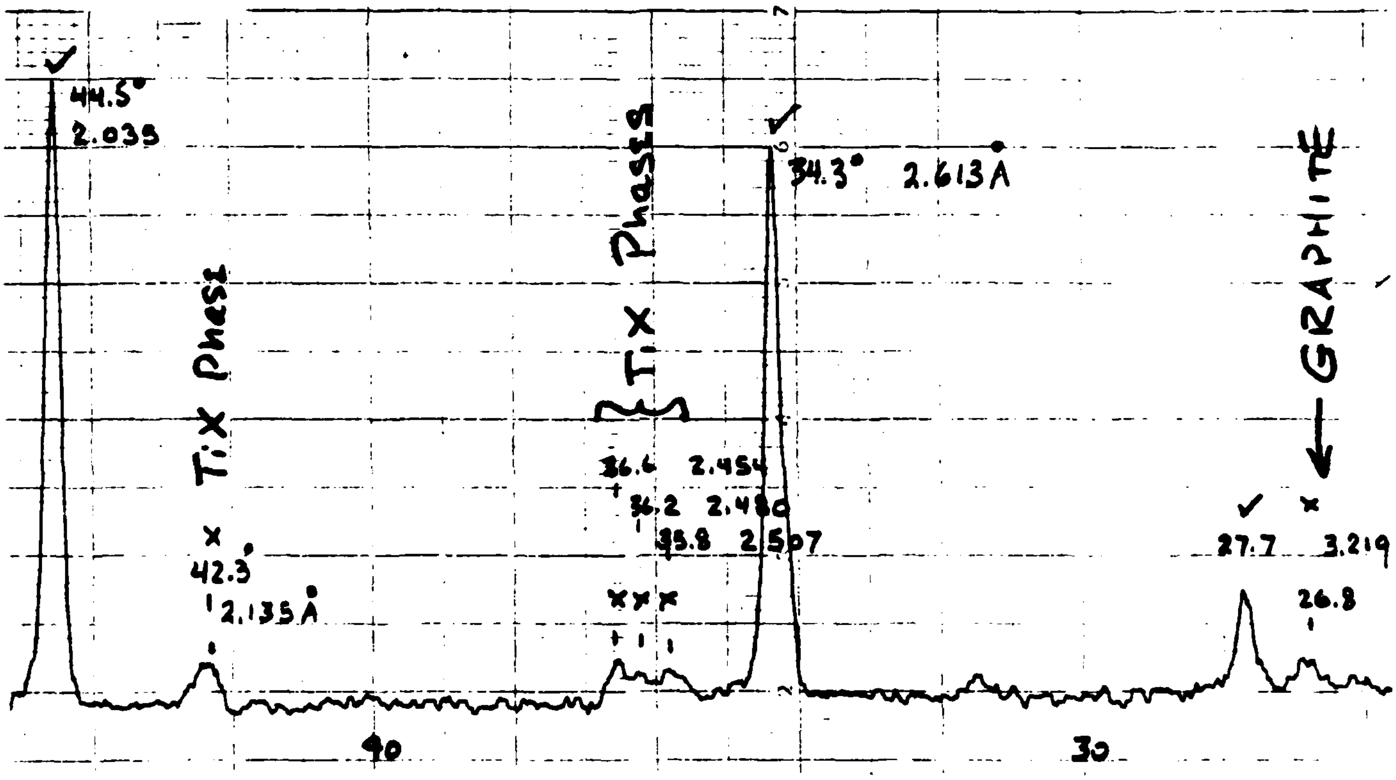
Manufacturer	Lot/ART NB Reference
A.E.E.	VII - 137
Cerac	VII - 108
ESK	VIII - 15
H. C. Starck	VI - 139
	VI - 150
	VII - 114
	VII - 115
	VIII - 20
KBI	VII - 116
UCC	VIII - 77

ANALYSIS OF REACTANTS

Material	TiB_2
Supplier	Atlantic Equipment Engineers
Product Designation	Ti-275, -325 mesh, 99.8%
ART Lot No.	N.B. - VII-137
ART Anal. No.	1069

Ti	63.70 %	
B	(1)	
O	2.40	
C	1.56	
N	3.10	
Spectrographic	(1)	
X-ray	$\frac{I2\theta \cong 45°}{I2\theta \cong 42°}$	(2)
Surface area	m^2/g	
Particle size	MV	μm
(microtrac)	PH	
	PM	
	PS	

( ) Foot notes summarized at end of Appendix C

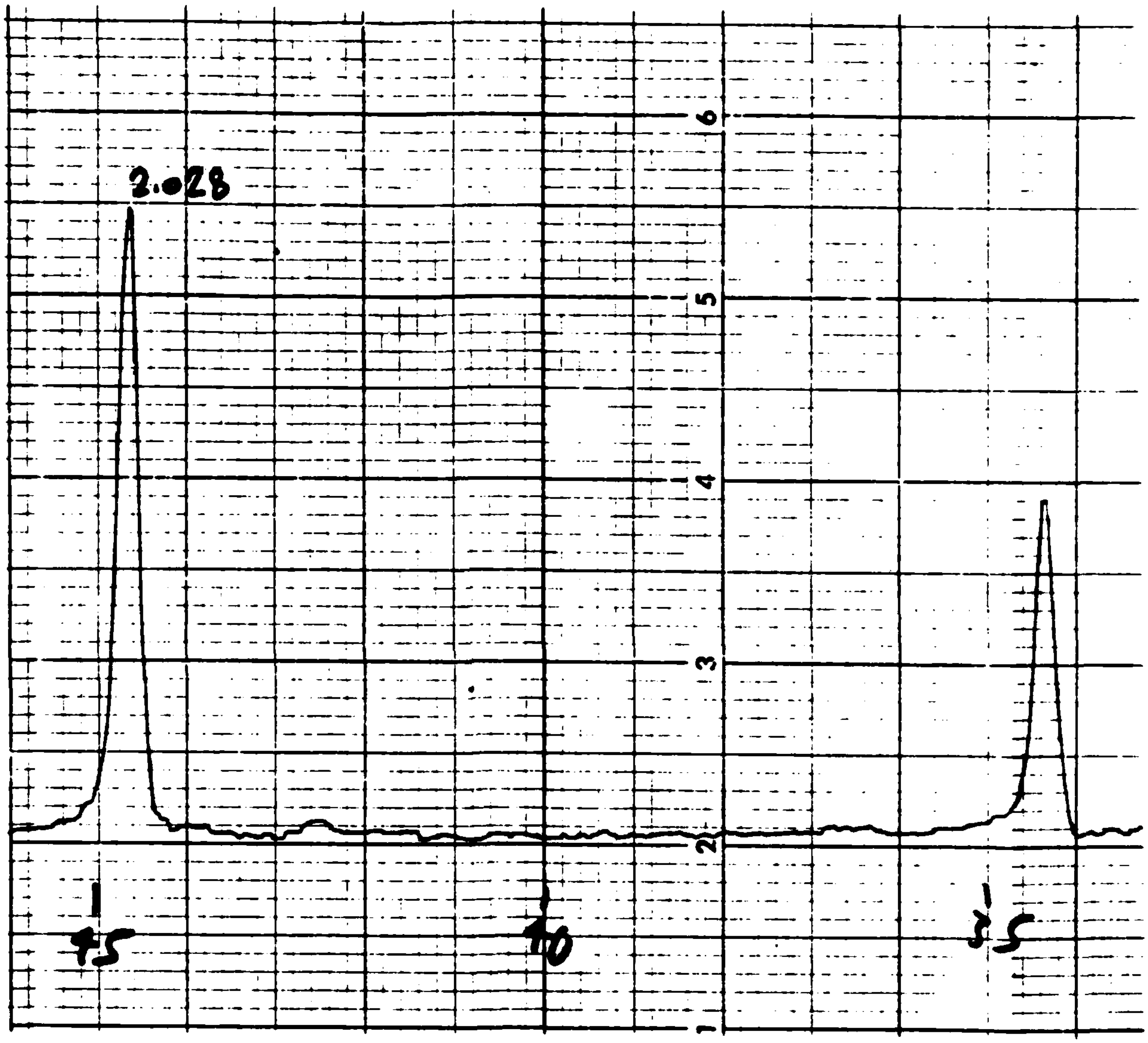


X-ray Diffraction Pattern of TiB_2 (Atlantic Equipment Engineers, VII-137)

ANALYSIS OF REACTANTS

Material	TiB_2
Supplier	Cerac
Product Designation	T.1221; -150 +325 mesh; 99.5 purity
ART Lot No.	N.B. - VII-108
ART Anal. No.	1000
Ti	69.31 %
B	27.44
O	0.74
C	0.58
N	0.19
Spectrographic	
Ca	0.1 %
Fe	0.06
Cr, Si	0.02
Others	< 0.01
X-ray	$\frac{34}{1}$
Surface area	0.61 m^2/g.
Particle size	MV 70 microns
	PH 117
	PM 68
	PS 14

( ) Foot notes summarized at end of Appendix C

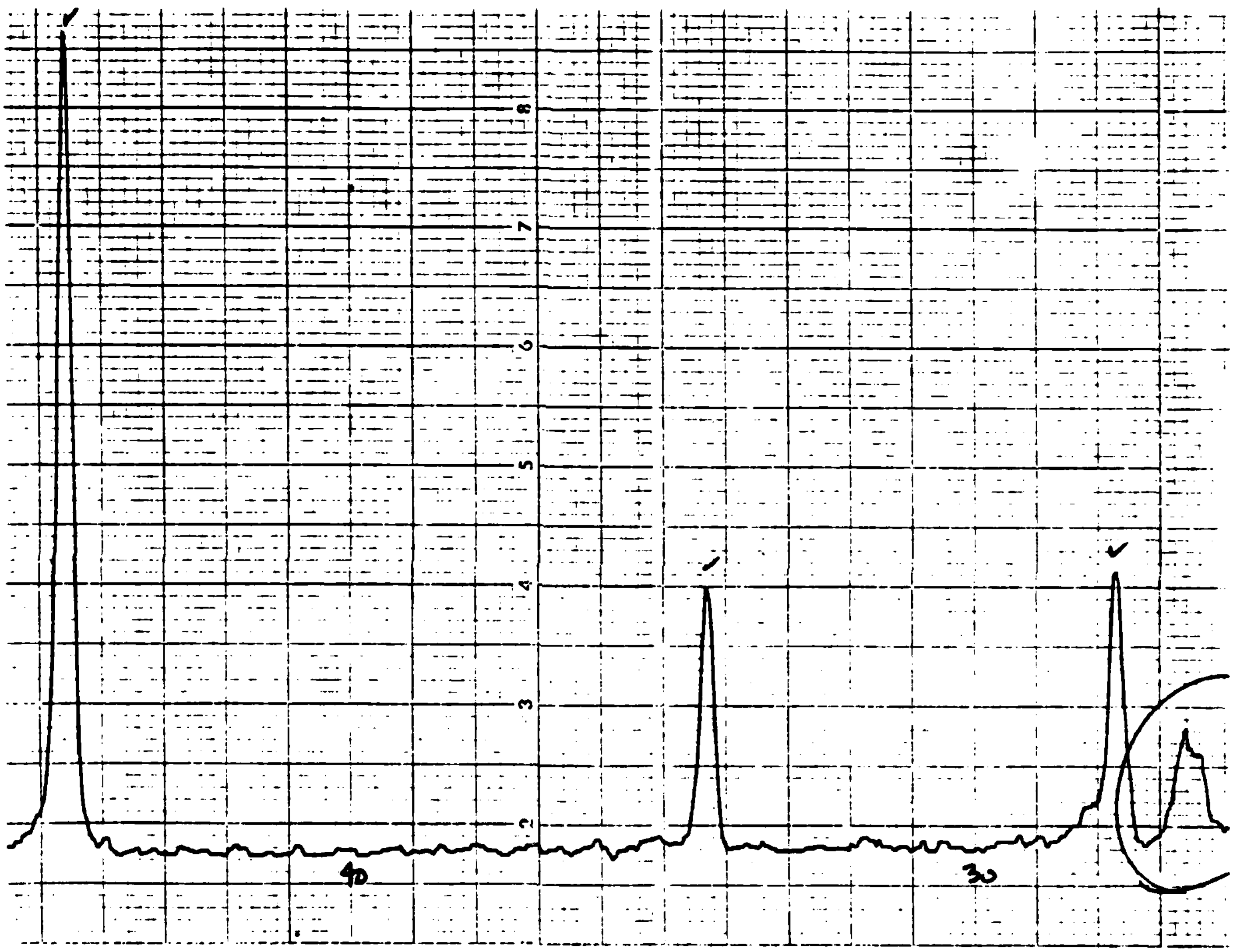


X-ray Diffraction Powder Pattern for TiB_2 (Cerac, VII-108)

ANALYSIS OF REACTANTS

Material		TiB_2	
Supplier		E.S.K.	
Product Designation		(3)	
ART Lot No.		N.B. - VIII - 15	
ART Anal. No.		1105	
Ti		66.56 %	69.3 (4) (5)
B		26.32	
O		0.77	
C		1.65	
N		0.04	
Spectrographic			
Ca		0.2 %	
W, Fe		0.02	
Si		0.01	
Others		< 0.01	
X-ray		$\frac{70}{1}$	(6)
Microtrac	MV	13 microns	
	PH	24	
	PM	11	
	PS	4.2	

( ) Foot notes summarized at end of Appendix C



X-ray Diffraction Powder Pattern for TiB_2 (E.S.K., VIII-15)

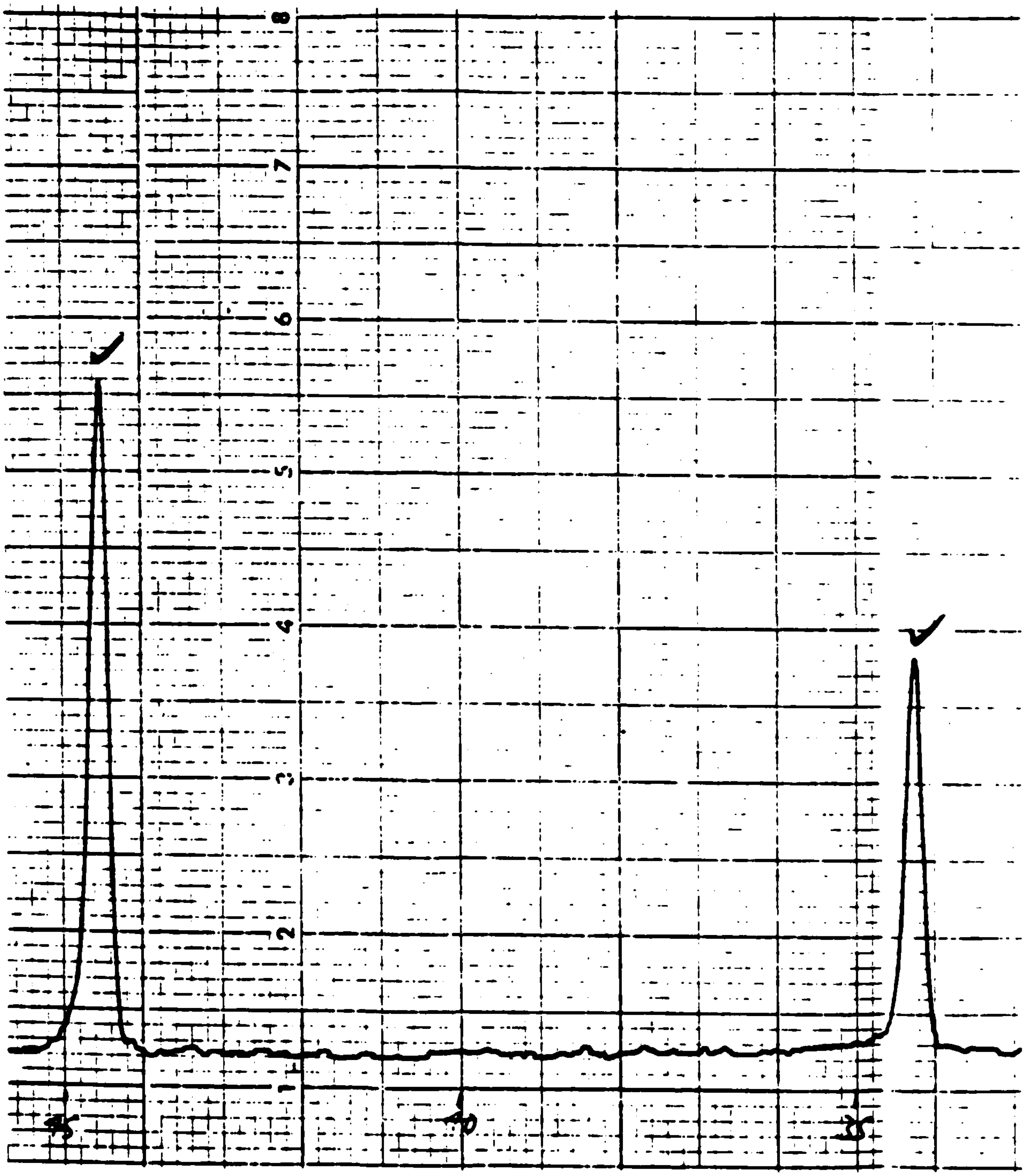
ANALYSIS OF REACTANTS

Material	TiB_2
Supplier	KBI
Product Designation	-325 mesh
ART Lot No.	NB - VII-116
ART Anal. No.	1002

Ti	69.70 %
B	30.10
O	0.45
C	0.51
N	0.008
Spectrographic	
All	< 0.1 %

Surface area	0.61 m^2/g	
X-ray	$\frac{44}{0}$	
Particle size	MV	14 microns
	PH	25
	PM	12
	PS	5.1

( ) Foot notes summarized at end of Appendix C



X-ray Diffraction Powder Pattern for TiB_2 (KBI, Ref. VII-116)

ANALYSIS OF REACTANTS

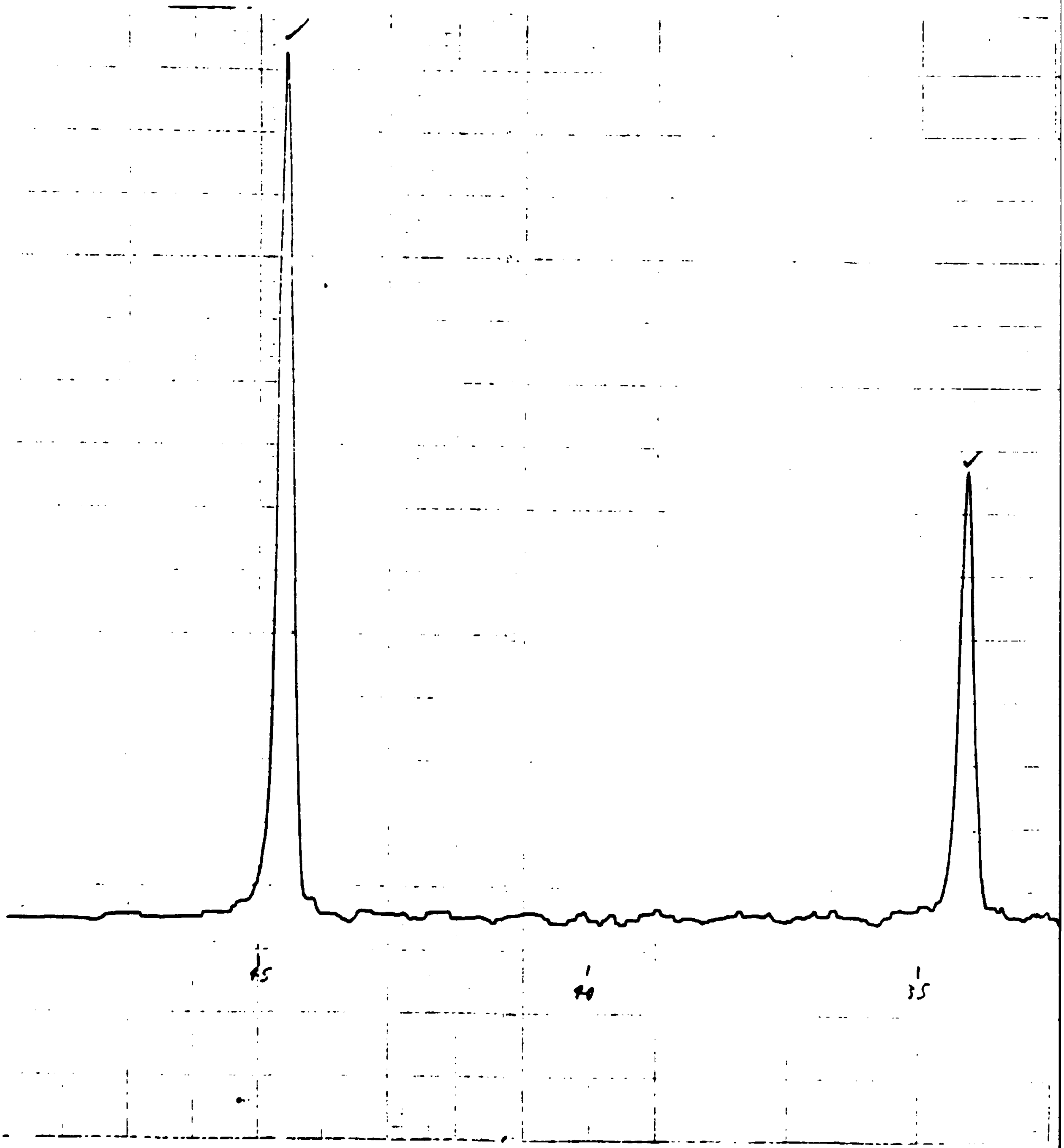
Material	TiB_2
Supplier	KBI
Product Designation	Lot HOS1, -325 mesh
ART Lot No.	NB - VIII-9
ART Anal. No.	1104

Ti	70.34 %	69.5 (4)	68.35 (7)
B	29.50		29.50 (7)
O	.74		.57 (7)
C	.34		.30 (7)
N	.06		.09 (7)
Spectrographic			
Fe	.04 %		.09
Ca	.1		
W	.06		

X-ray $\frac{68}{0}$

Particle size	MV	10 microns
	PH	20
	PM	8.0
	PS	3.3

( ) Foot notes summarized at end of Appendix C



X-ray Diffraction Powder Pattern for TiB_2 (KBI, VIII-9)

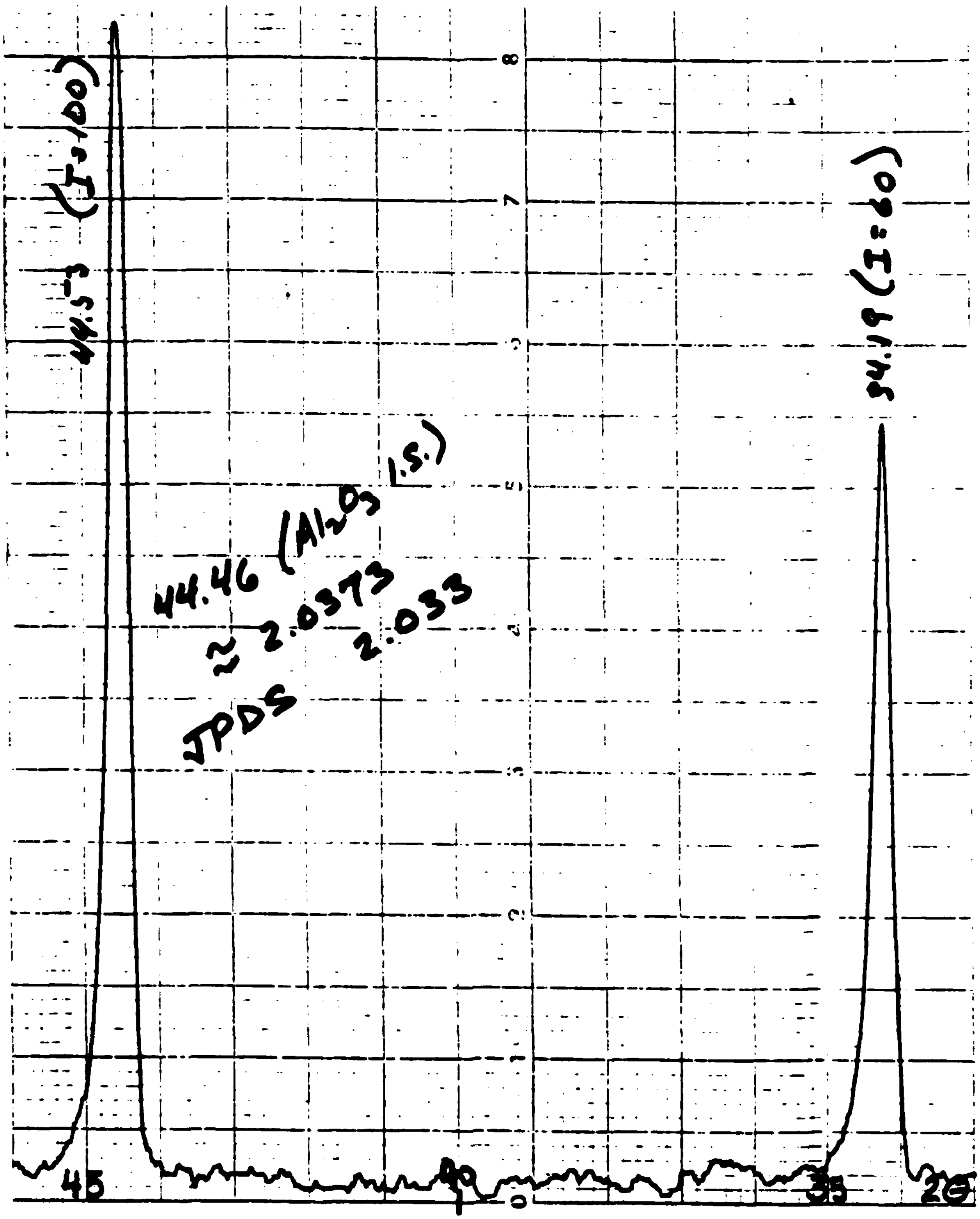
ANALYSIS OF REACTANTS

Material	TiB_2
Supplier	H. C. Starck
Product Designation	(9) (10)
ART Lot No.	NB - VI-139
ART Anal. No.	696

Ti	68.18	%
B	30.04	
O	0.25	
C	0.14	
N	0.097	
Spectrographic		
Si, W	0.02	%
Cr, Fe	0.01	
Others	< 0.01	
X-ray	$\frac{80}{0}$	

Particle size	MV	22 microns
	PH	41
	PM	18
	PS	5.4

( ) Foot notes summarized at end of Appendix C



X-ray Diffraction Powder Pattern for TiB_2 (H. C. Starck, Ref. VI-139

ANALYSIS OF REACTANTS

Material	TiB_2
Supplier	H. C. Starck
Product Designation	(9, 11)
ART Lot No.	NB - VI-150
ART Anal. No.	734

Ti	69.92	%
B	29.65	
O	0.54	
C	0.16	
N	0.24	

Particle size	MV	9.9	microns
	PH	19	
	PM	8.2	
	PS	3.1	

( ) Foot notes summarized at end of Appendix

ANALYSIS OF REACTANTS

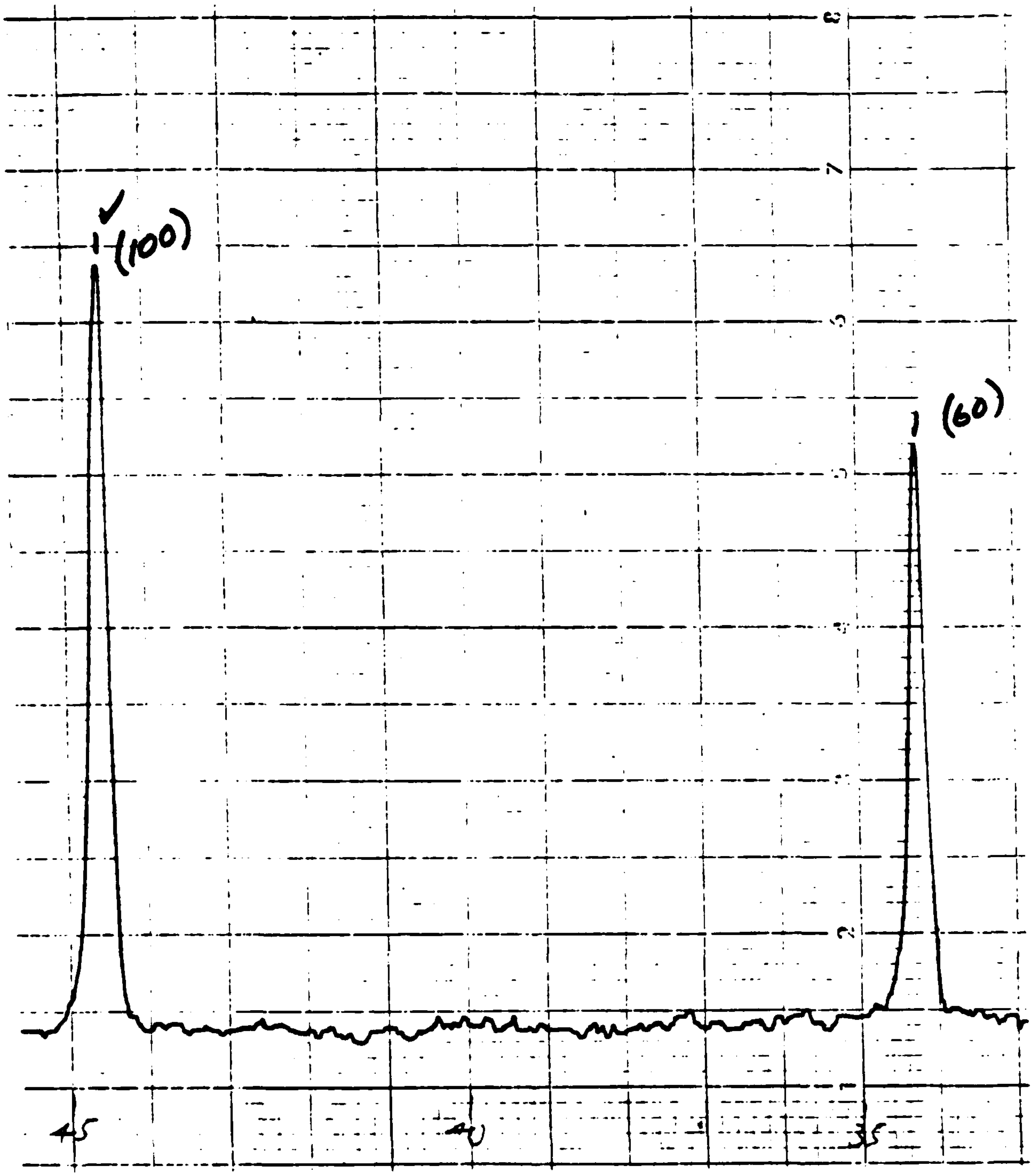
Material	TiB_2
Supplier	H. C. Starck
Product Designation	Lot 2388 (9, 10)
ART Lot No.	NB - VII-114
ART Anal. No.	1002

Ti	68.50 %	
B	23.51	30.97 (7)
O	0.19	0.2 (7)
C	0.44	0.1 (7)
N	0.006	0.2 (1)

Surface area 0.56 m^2/g

Particle size	MV	26 microns
	PH	46
	PM	22
	PS	6.5

( ) Foot notes summarized at end of Appendix C



X-ray Diffraction Powder Pattern for TiB_2 (H. C. Starck, Ref. VII-114)

ANALYSIS OF REACTANTS

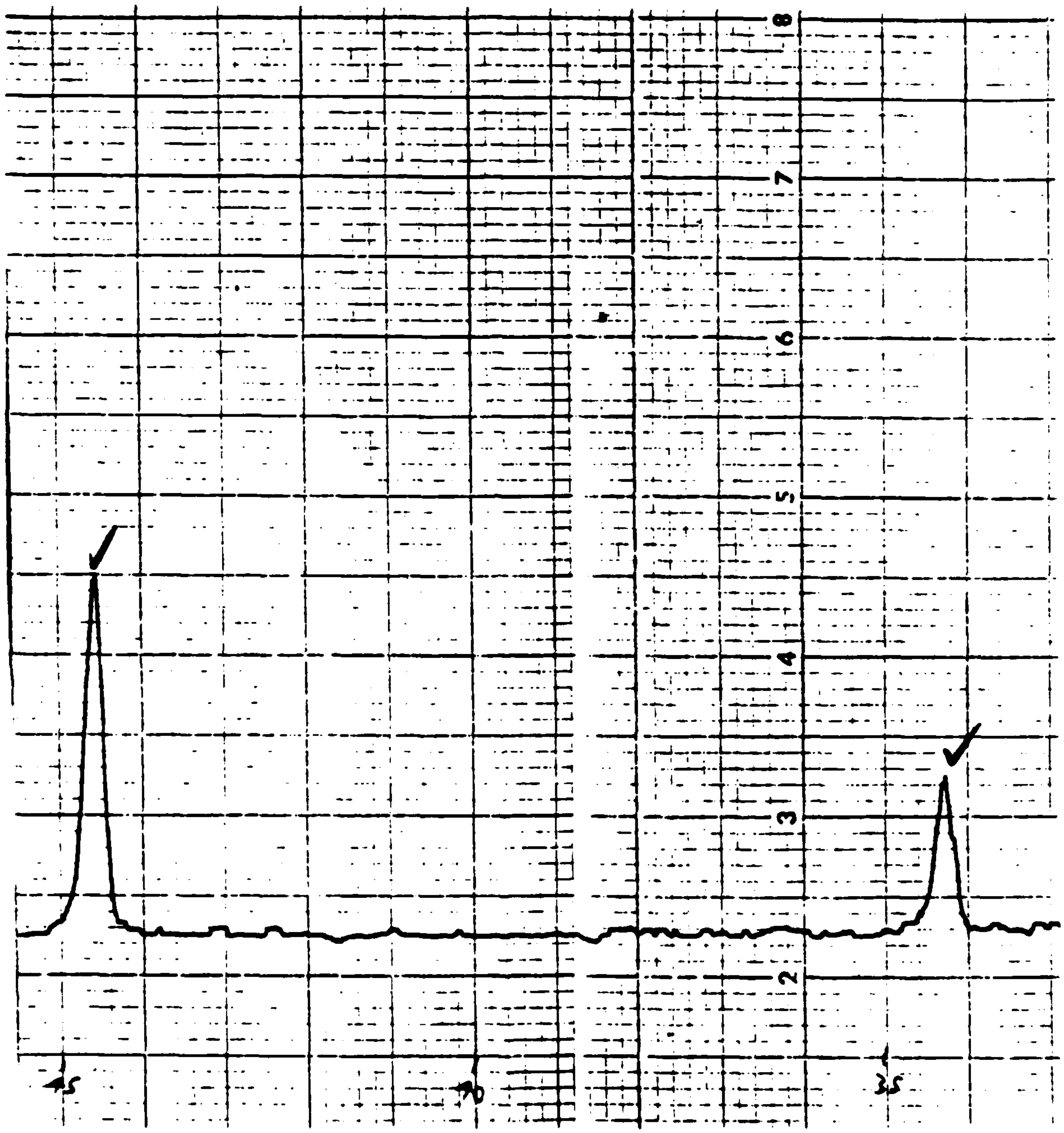
Material	TiB_2
Supplier	H. C. Starck
Product Designation	(9, 11)
ART Lot No.	NB - VII-115
ART Anal. No.	1002

Ti	69.26 %	
B	25.52	30.2 (7)
O	0.19	0.19 (7)
C	0.52	0.3 (7)
N	0.007	0.09 (7)

Surface area 0.67 m^2/g

Particle size	MV	24	microns
	PH	46	
	PM	20	
	PS	7.9	

( ) Foot notes summarized at end of Appendix C



X-Ray Diffraction Powder Pattern for TiB_2 (H. C. Starck, Ref. VII-115)

ANALYSIS OF REACTANTS

Material	TiB_2
Supplier	H. C. Starck
Product Designation	Grade A - Lot S-4955
ART Lot No.	831028, VIII-20
ART Anal. No.	1120

Ti	69.86 %
	73.7
B	22.65
O	1.59
C	0.15
N	0.17

Spectrographic	
Ca	0.2 %
W	0.06
Others	≤ 0.02

Surface Area 1.87 m^2/g

Particle size	MV	4.7 microns
	PH	7.4
	PM	4.3
	PS	2.4

( ) Foot notes summarized at end of Appendix C

X-ray Diffraction Powder Pattern for TiB_2 (HCST, VIII-20)

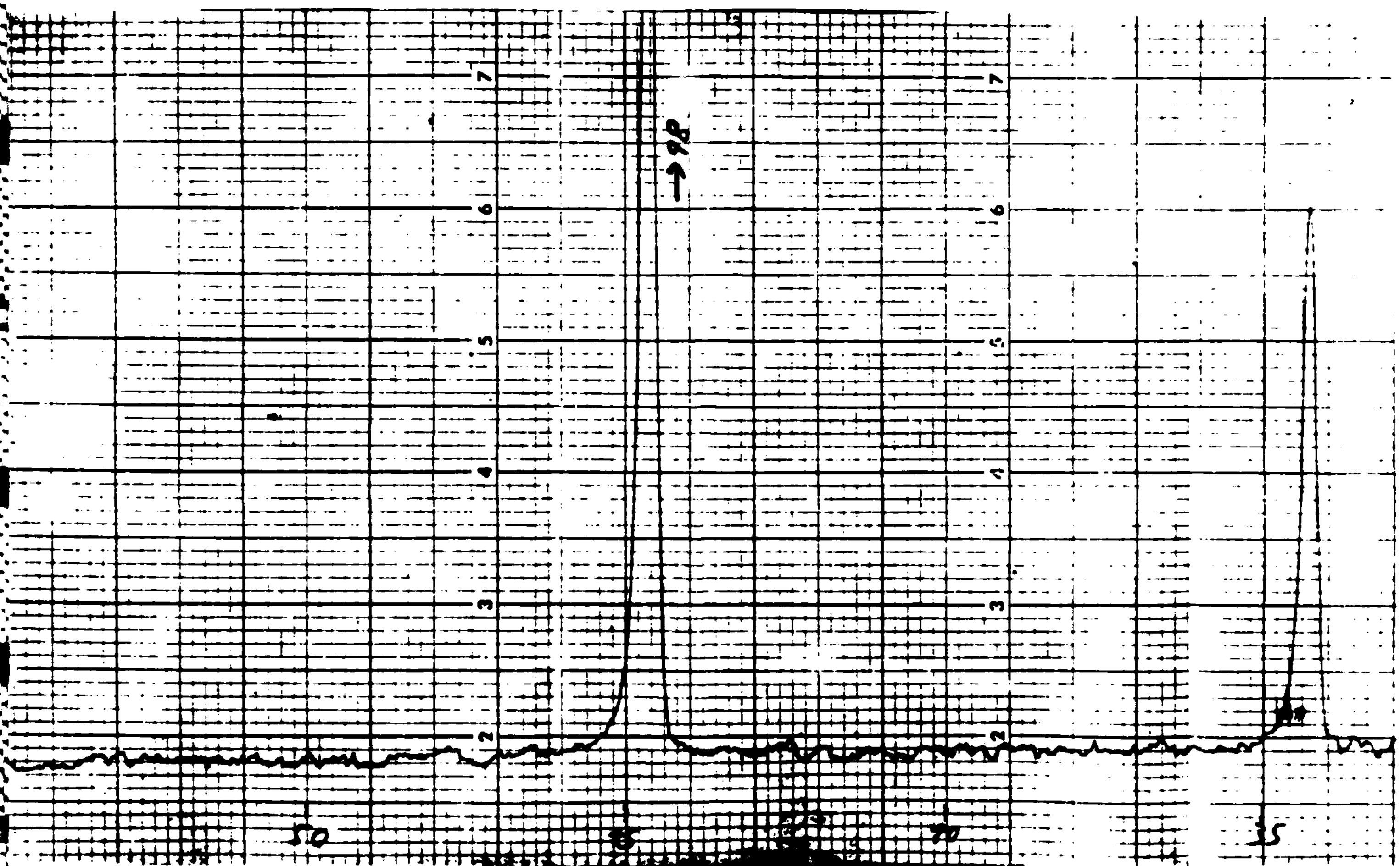
ANALYSIS OF REACTANTS

Material	TiB_2
Supplier	Union Carbide
Product Designation	-325 mesh, 98 + pure
ART Lot No.	NB - VIII-77
ART Anal. No.	C-197

Ti	68.82, %	73.00 (8)	71.87 (4)
B	30.46		
O	0.61	0.73	
C	0.45	0.47	
N	0.22	0.22	
Spectrographic			
Ca	0.4 %	0.2	
Fe	0.06	0.06	
Si	0.04	0.06	
W	0.02	< 0.01	
Others	< 0.01		

Particle Size	MV	9.8 microns
	PH	19
	PM	7.7
	PS	3.0

( ) Foot notes summarized at end of Appendix C



X-ray Diffraction Powder Pattern for TiB_2 (UCC VIII-77)

FOOTNOTES FOR APPENDIX C

(1) This material so far from pure, analyses was not completed.

(2) Four separate impurity peaks shown.

(3) Purest available -325 mesh powder requested.

(4) SUNY - Buffalo, X-ray Fluorescence

(5) Unless specified, Ti determined by Buffalo Testing Laboratories colormetrically

(6) Major impurity peak observed, 3.37 Å, presumably graphite.

(7) Data on accompanying certification sheets.

(8) Sample resubmitted for relicate analyses.

(9) Samples supplied by a laboratory, studying TiB_2 for cathodes for Hall Cells.

(10) Yielded good test results in the Hall Cell test.

(11) Yielded unacceptable test results in the Hall Cell tests.

APPENDIX D

ENGINEERING ESTIMATES FOR

LARGE SCALE TITANIUM DIBORIDE PRODUCTION FACILITY

SECTION C - 2.3

QUANTITATIVE COST COMPARISON

The following is a description of the estimated costs for producing 100 tons/year of ultrafine titanium diboride powder utilizing ART's formation technology.

SUMMARY

Scope:

A full-scale titanium diboride powder production plant capable of 100 tons/year minimum output.

Facility Requirements:	- 5,000 square feet
	- 20' clearance
	- 500 KVA power - 480 3Ø
	- cooling water - deionized
	- bulk argon tank
Capital Equipment Expense:	$751,000 plus installation
Capacity (annual):	838,656 lbs. (theoretical)
	300,000 lbs. (likely)
Cost of Production:	\$4-5/lb. of TiB_2 plus labor

FACILITY REQUIREMENTS

Space: Approximately 5,000 square feet of open, standard butler type construction with 20' ceiling clearance. Concrete floor - no special reinforcement required.

Services: Electrical - 500 KVA of 3 phase 480V power would be optimal for normal, balanced loads. Additional 250 KVA would be required if all 4 modules were running simultaneously.

Water - A recirculating cooling system with deionized water is required. Flow capacity of 150 GPM.

Argon - Bulk tank - 50,000# liquid argon

A railcar siding may be desirable, as 20 tons of raw material are required monthly.

CAPITAL EQUIPMENT

Material Handling	
- Fork truck, steel hoppers, bins, etc.	$40,000
Mix Preparation	
- Pelletizers and/or spray driers	25,000
Calcining	
- Forced air calciners	36,000
Reactor Modules (4)	
- ART Proprietary furnaces	600,000
Deagglomerating	
- Screeners, mills, pulverizers	50,000
TOTAL (excludes installation)	$751,000

CAPACITY

Requirement: 200,000 lbs./year

Assumptions: 6,240 hrs. production/year

Reactors have been operated continuously at production rates of 24/lbs./hr. Optimal product is achieved at 12/lbs./hr. rate.

Theoretically (100% utilization, 24 lbs./hr., 7 day week) output could be:

4 units x 24 lbs./hr. x 24 hrs./day x 7 days/wk. x 52 wks./yr. or 838,656 lbs/yr.

The most likely production scenario, however is as follows:

4 units x 12 lbs./hr. x 120 hrs./wk. x 52 wks./yr. or 299,520 lbs./yr.

This exceeds the requirement by 50%.

ECONOMICS - 200,000 lbs.

Preliminary Estimate:

Material Costs			
230,000 lbs.	TiO_2 @ \$0.75		\$172,500
79,000 lbs.	B_4C @ \$6.00		474,000
52,000 lbs.	carbon @ \$0.40		20,800
			\$667,300
Electrical			
6500 mwh	@ \$60		\$ 90,000
Argon			50,000
Supplies, Consumptibles, etc.			60,000
		Sub-total	\$867,300
			\$4.32/lb.
Depreciation			0.54/lb.
			\$4.86/lb.
Labor			
Supervisory (1)	\$ 35,000		
Direct (9)	180,000		
Fringes/benefits	43,000		
	\$258,000		\$1.29/lb.
			\$6.15/lb.

COMPARISON

Assuming a 30% selling margin for ART, the relative pricing is as follows:

Supplier	Price per Pound	(% ART's price)
ART	\$ 8.79	100
Electroschmelzwerk Kemplin	\$18.15	206
Union Carbide	\$33.00	375
Kawecki Berylco	\$33.00	375
H. C. Starck	\$37.50	427
Atlantic Equipment Engineers	\$63.12	718

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A PROCESS STUDY FOR MANUFACTURING ULTRA-FINE TITANIUM DIBORIDE POWDER
Peter T. B. Shaffer
Advanced Refractory Technologies, Inc.
699 Hertel Avenue
Buffalo, NY 14207

Technical Report MTL TR 86-18, May 1986, 133 pp - illus-tbls, Contract DAAG46-83-C-0171
D/A Project 1L263102D071
Final Report, 3/26/83 to 6/25/84

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Key Words

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Synthesis chemistry
High temperature

Advanced Refractory Technologies, Inc., of Buffalo, NY has demonstrated the feasibility of utilizing a rotary high temperature carbon tube furnace to form reasonably pure titanium diboride powder. The reaction used was the carbothermic reduction of boron carbide in the presence of highly reactive titanium dioxide. The resultant titanium diboride powder was characterized by X-ray diffraction analysis and microscopic observation. The powder possessed a mean specific surface area of 0.5 square meter per gram. The rate of powder formation through use of the rotary kiln process was approximately five kilograms per hour. Experimental trials to form fully dense titanium diboride by hot pressing were unsuccessful. Further developmental work utilizing the rotary kiln process to attempt to achieve stoichiometric titanium diboride powder of the required purity for successful hot pressing is indicated.

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